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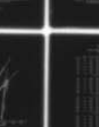
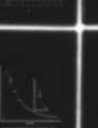
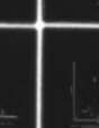
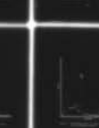
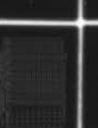
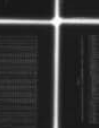
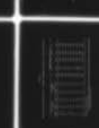
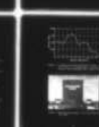
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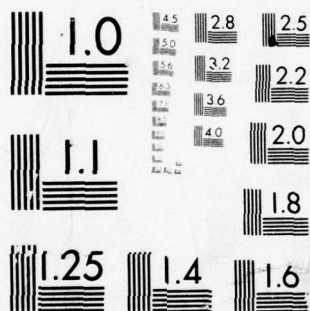
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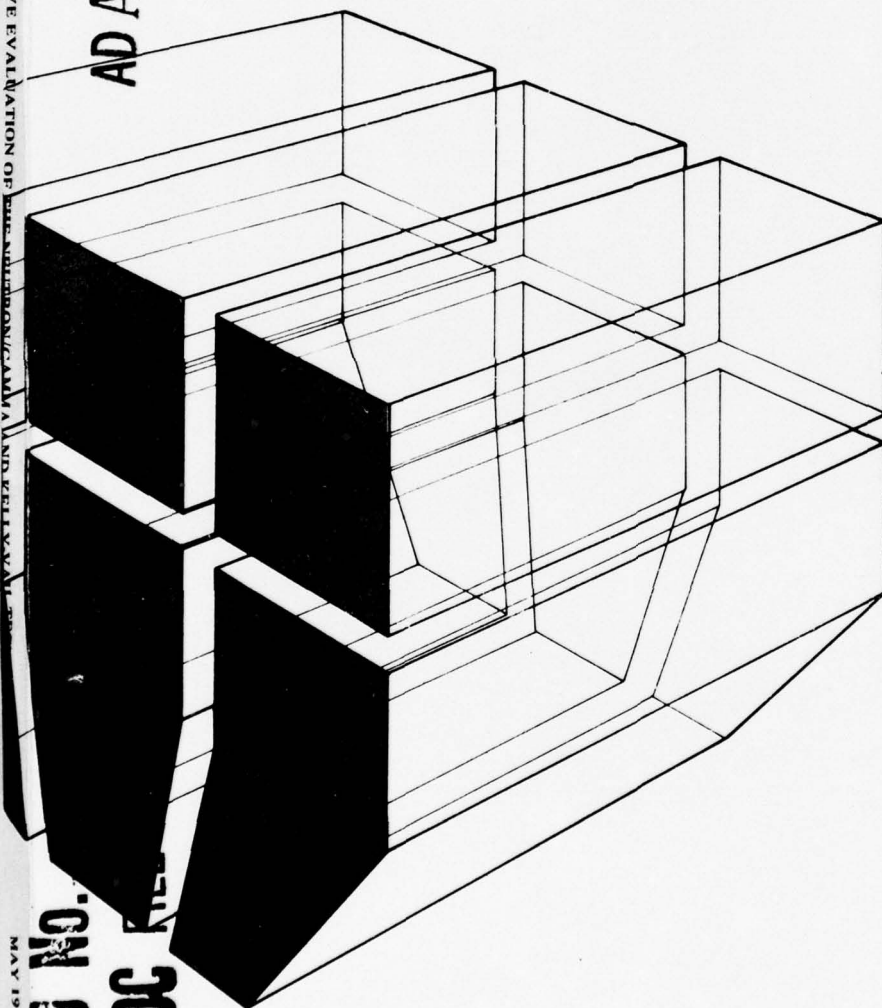
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A COMPARATIVE EVALUATION OF THE NEUTRON/GAMMA  
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A COMPARATIVE EVALUATION OF THE NEUTRON/GAMMA AND KELLY-VAIL TECHNIQUES FOR DETERMINING WATER AND CEMENT CONTENT OF FRESH CONCRETE



by  
P. A. Howdyshe



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4. TITLE (and Subtitle) A COMPARATIVE EVALUATION OF THE NEUTRON/GAMMA AND KELLY-VAIL TECHNIQUES FOR DETERMINING WATER AND CEMENT CONTENT OF FRESH CONCRETE.		5. TYPE OF REPORT & PERIOD COVERED FINAL rept.
7. AUTHOR(s) 10 P. A. Howdyshe11		6. PERFORMING ORG. REPORT NUMBER
9. PERFORMING ORGANIZATION NAME AND ADDRESS CONSTRUCTION ENGINEERING RESEARCH LABORATORY P.O. Box 4005 Champaign, Illinois 61820		8. CONTRACT OR GRANT NUMBER(s) 16
11. CONTROLLING OFFICE NAME AND ADDRESS		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS 4A762719AT41-T4-008
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		12. REPORT DATE May 1977
		13. NUMBER OF PAGES 166 167 P.
		15. SECURITY CLASS. (of this report) UNCLASSIFIED
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited.		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
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19. KEY WORDS (Continue on reverse side if necessary and identify by block number)  neutron/gamma Kelly-Vail mortar test series concrete test series		
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The evaluation consisted of two major experimental test series: (1) a mortar test series, which was used to evaluate and develop calibration methodology for the neutron/gamma system, and (2) a concrete test series, which directly compared the chemical and neutron/gamma systems and evaluated their accuracies.

Test results indicate that the neutron/gamma method can estimate water contents to  $\pm 6$  percent, and cement contents to  $\pm 9$  to 22 percent, depending on the type of aggregate used. This compared to cement and water content accuracies of 7.1 and 5.2 percent, respectively, for the chemical technique. The results also indicated that the chemical system was a better estimator of concrete strength potential than the neutron/gamma system.

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## FOREWORD

Research for this project was performed for the Office of the Chief of Engineers (OCE), Directorate of Military Construction, under Project 4A762719AT41, "Design, Construction, and Operations and Maintenance Technology for Military Facilities"; Task T4, "Construction Systems Technology"; Work Unit 008, "Rapid Testing--Plastic PCC." The applicable QCR number is 1.06.003. Mr. R. Liebhardt was the OCE Technical Monitor.

The research was conducted by the Construction Materials Branch (MSC), Materials and Science Division (MS) of the U.S. Army Construction Engineering Research Laboratory (CERL). Mr. P. Howdysheill, Chief of MSC, was the CERL Principal Investigator.

Dr. G. Williamson is Chief of MS. COL J. E. Hays is Commander and Director of CERL, and Dr. L. R. Shaffer is Technical Director.

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A COMPARATIVE EVALUATION OF THE  
NEUTRON/GAMMA AND KELLY-VAIL  
TECHNIQUES FOR DETERMINING WATER  
AND CEMENT CONTENT OF FRESH CONCRETE

1 INTRODUCTION

Background

The inspection and testing procedures currently used to evaluate the quality of fresh concrete in the field are slump, unit weight, and air entrainment tests. The properties of hardened concrete are determined by breaking compression and flexural test samples. Although these procedures are generally accepted and have not changed significantly during the past several years, they have several deficiencies.

The most critical deficiency is the inherent time lag between concrete placement and the evaluation of hardened concrete quality. This lag, which stems from the curing time required for the compressive and/or flexural test samples, usually varies from 3 to 28 days. Even for the accelerated curing techniques, the lag time is 24 or more hours, depending on the method of curing. The lag time makes the replacement of substandard concrete expensive and difficult, since it allows the concrete to set and harden before its quality can be ascertained.

Another major deficiency of current testing procedures is their failure to relate directly to either the material or construction parameters that influence concrete quality. For example, the slump test--often erroneously considered a measure of workability--is basically a means of detecting variations in the uniformity of a mix of given nominal proportions. Also, the strength of compressive and/or flexural test samples does not automatically indicate the strength of in-situ concrete, since curing and placing procedures differ.

In an attempt to improve current procedures, several different concrete tests have been proposed. One is the rapid field determination of fresh concrete constituent material (aggregates, cement, water, and various additives and admixtures) proportions. Such a test, if sufficiently accurate, could be an excellent indication of the quality potential of a fresh concrete (assuming that the constituents themselves were of acceptable quality). The test could be used to evaluate mixer efficiency and determine concrete homogeneity, and to determine mix proportions of homogeneous mixes. Since the water/cement ratio for a specific cement type is the primary parameter influencing its potential strength, such a method, if sufficiently accurate and rapid, should be able to estimate strength potential prior to the

setting and hardening of concrete in the forms. There are two techniques for determining concrete constituent material proportions. One is a new, innovative neutron/gamma system that relies on a characteristic energy emission of various elements (multiple signature method) to determine constituent material proportions. The other is a chemical technique that relies on chloride ion titration to determine water content and flame photometry to determine cement content. Hereafter, the chemical technique will be referred to as the Kelly-Vail technique.\*

### Objective

The objective of this investigation is to evaluate the two techniques for determining concrete constituent material proportions, to evaluate the accuracies of both systems, and to determine their abilities to estimate concrete strength potential.

### Approach

The evaluation consisted of two major experimental test series: (1) a mortar test series, which was used to evaluate and develop calibration methodology for the neutron/gamma system; and (2) a concrete test series which directly compared the Kelly-Vail and neutron/gamma systems and evaluated their accuracies.

---

\* R. T. Kelly and J. W. Vail developed this technique.



## 2 RELATED INVESTIGATIONS

The advantages of developing a method for rapidly determining fresh concrete constituent material proportions have been apparent for many years. With only one major exception (electrical resistance), the proposed methods for determining constituent material proportions fall into two broad categories: chemical-mechanical or nuclear. (The bibliography lists proposed methods for determining water and cement content of fresh concrete.)

The multiple signature concept for determining concrete constituents was conceived as a solution to the deficiencies of the non-unique, single-signature methods for determining water and/or cement contents of fresh concrete. Phase I (equipment feasibility) of a two-phase study was initiated in April 1972 and completed in December 1973.<sup>1</sup> The results indicated that no single neutron source technique was capable of obtaining quantitative signatures on the four primary elements (H, C, Si, and Ca)\* requested, but that it was possible to combine neutron source techniques to meet the analytical requirements of the multiple signature concept. Based on these results, a laboratory prototype instrument was constructed (Phase II), and instrument evaluation was initiated in late 1974.

One paper has been presented on results obtained from preliminary laboratory tests on portland cement mortars.<sup>2</sup> Major results were:

1. In an 11-min test, the prototype neutron gamma system could obtain quantitative signatures on the H, Ca, Si, and C present in fresh concrete. The accuracy (counting error) of the unit was 0.5 to 2.0 percent for the H, Ca, and Si signatures, and 2.0 to 4.0 percent for the C signature.
2. The Si, Ca, and C signatures are sensitive to the presence of H in the sample; however, within the water content variations normally found in concrete, this sensitivity was small.
3. The H signature background channels were overriding a nearby Ca peak, thus allowing the Ca content of the sample to interfere with the background count of the H signature.

<sup>1</sup> M. C. Taylor, *A New Method for Field Analysis of Plastic Concrete--Feasibility Study*, Technical Report M-64/AD#771908 (U.S. Army Construction Engineering Research Laboratory [CERL], December 1973).

\* Hydrogen, carbon, silicon, and calcium.

<sup>2</sup> P. A. Howdysshell, "Preliminary Evaluation of the Neutron/Gamma Technique to Determine the Water and Cement Content of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (CERL, May 1975).

4. Tests on mortar constituents (cement and sand) indicated that the signatures obtained from constituent tests cannot be used directly to estimate the signatures for the mortar mixes due to the drastic differences in matrix compositions (presence of water).

The Kelly-Vail technique was originally suggested by Chaplin and Kelly<sup>3</sup> in 1967 and described in detail by Kelly and Vail<sup>4</sup> in 1968. Their results indicated that the method was rapid (less than 15 min for both cement and water content) and accurate (water content  $\pm 4.1$  percent and cement content  $\pm 4.6$  percent).

More recently, the U.S. Army Construction Engineering Research Laboratory (CERL) has extensively tested and evaluated the Kelly-Vail system. As a result of the extensive program, four separate papers have been presented on the topic. The initial paper indicated that the procedure can rapidly (approximately 15 min) determine the water and cement content of fresh concrete and that it can be used to predict strength potential with an accuracy equal to that of predicting strength from known mix proportions.<sup>5</sup> Aggregate type (calcareous fines) was the only major concrete parameter that significantly influenced test results. Influences of aggregate moisture condition, mix proportions, and length of mixing time were minor.

The second paper indicated that the system could be housed in a pick-up truck camper shell in a ready-to-use configuration.<sup>6</sup> Early field tests demonstrated the mobility and field worthiness of the technique.

The third paper evaluated the relation between Kelly-Vail test results and the strength potential of fresh concrete.<sup>7</sup> It was concluded that for a given cement type, air entrainment was the only major material parameter other than water/cement ratio that influenced concrete strength over a normal range of aggregate types and sizes. Thus, the Kelly-Vail test, when used in conjunction with air content measuring devices, provides a rapid means for determining the strength potential of fresh concrete.

<sup>3</sup> C. A. Chaplin and R. T. Kelly, "The Analysis of Concrete," *Chemistry and Industry* (2 September 1967), pp 1467-1473.

<sup>4</sup> R. T. Kelly and J. W. Vail, "Rapid Analysis of Fresh Concrete," *Concrete* (April and May 1968) pp 140-146, 206-210.

<sup>5</sup> P. A. Howdysshell, *Laboratory Evaluation of a Chemical Technique to Determine Water and Cement Content of Fresh Concrete*, Interim Report M-97/AD#784055 (CERL, July 1975).

<sup>6</sup> P. A. Howdysshell, *Evaluation of a Chemical Technique to Determine Water and Cement Content of Fresh Concrete*, Technical Manuscript M-119/ADA005576 (CERL, January 1975).

<sup>7</sup> P. A. Howdysshell, "Correlating Kelly-Vail Test Results to the Strength Potential of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (CERL, May 1975).

The most recent paper about the Kelly-Vail system was an Operations Guide that outlined required equipment and methods of operation.<sup>8</sup>

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<sup>8</sup> P. A. Howdysshell, *Operations Guide--Water and Cement Content of Fresh Concrete*, Technical Report M-177/ADA022697 (CERL, September 1975).

### 3 DESCRIPTION OF NEUTRON/GAMMA AND KELLY-VAIL TECHNIQUES

#### Neutron/Gamma Technique

##### *Signature Element Concept*

The neutron/gamma technique is based on a multiple signature concept for determining concrete constituent material proportions. The concept uses the relationship between signature intensities and chemical element concentrations to obtain a quantitative analysis of select elements in a concrete sample. Assuming that the relationship between signature intensities and chemical element concentrations is linear, the constituent proportions of the sample can then be determined by linear simultaneous equations of the following form:

$$N_i = \sum_j k_{ij} C_j \quad [\text{Eq } 1]$$

where  $N_i$  = concentration of signature element "i" in the mix

$C_j$  = concentration of constituent "j" in the mix

$k_{ij}$  = concentration of signature element "i" in constituent "j"

To determine constituent material proportions, the number of signature elements must equal or exceed the number of chemically unique constituents; each constituent must contain at least one signature element; and the signature element concentrations in each constituent must be reasonably constant.

Signature element selection is dictated by: (1) the requirements of Eq 1; (2) a quantitatively detectable neutron/gamma signature in the elements; and (3) rapidity (less than 15 min) and accuracy of the entire procedure.

For concrete, Eq 1 requires a minimum of four signature elements, since it contains four chemically unique constituents (water, cement, fine aggregates, and coarse aggregates). For optimum accuracy and simplicity, signatures unique to each constituent should be used, with the unique signature having a high level of concentration in the constituent. For evaluating concrete constituents' chemical composition, water is  $H_2O$  with trace levels of dissolved and suspended materials; cements on an oxide basis are predominantly calcium with lesser quantities of Si, Al, Fe, S, Mg, Na, and  $K^*$  (see Table 1); and aggregates

---

\* Aluminum, iron, sulphur, magnesium, sodium, and potassium.



are normally either highly siliceous, calcareous, or some combination of the two. Many aggregates also contain significant, although relatively smaller quantities of Al, Fe, Mg, K, and Na.

Table 1

Typical Chemical Analysis of a Type I Portland Cement<sup>\*</sup>

Principal Oxides	Percent by Wt.	Other determinations	Percent by Wt.
SiO <sub>2</sub>	20.67	MgO	2.58
Al <sub>2</sub> O <sub>3</sub>	5.96	Na <sub>2</sub> O	0.12
Fe <sub>2</sub> O <sub>3</sub>	2.35	K <sub>2</sub> O	0.94
CaO	63.62	Loss of ignitions	1.37
SO <sub>3</sub>	2.13	Insoluble residue	0.26
		Free CaO	1.43

<sup>\*</sup> From G. E. Troxell, H. E. Davis, and J. W. Kelly, *Composition and Properties of Concrete*, 2nd ed. (McGraw-Hill, 1956, 1968), p 21.

When selecting signature elements, it should be noted that hydrogen which is nearly unique to water, is an obvious choice. Selecting the other three signature elements is more difficult. For highly siliceous aggregates, cement content is nearly proportional to the calcium in the mix. Conversely, for calcareous aggregates, cement content is nearly proportional to the silicon in the mix, and aggregate content is proportional to the carbon in the mix. Based on constituent composition, the four primary signature elements are hydrogen, calcium, silicon, and carbon.

Three different types of neutron/gamma interactions can be employed to obtain characteristic elemental signatures. These interactions are: (1) prompt gammas from neutron inelastic scattering; (2) prompt gammas from neutron capture; and (3) delayed gammas from the decay of the radioactive isotopes produced during neutron capture (conventional activation analysis).

The feasibility study conducted by Taylor<sup>9</sup> evaluated both the prompt and delayed gammas from three different neutron sources (Cf-252,

<sup>9</sup> M. C. Taylor, *A New Method for Field Analysis of Plastic Concrete --Feasibility Study*, Technical Report M-74/AD#771908 (CERL, December 1973).

Pu-Be, and a sealed tube 14-MeV neutron generator). Table 2 summarizes the results of the feasibility study for each of the primary signature elements as well as some minor elements. Not one of the single-source techniques could determine all four primary elements, but the results did indicate that a dual-source, multiple-analysis procedure could rapidly (less than 15 min) obtain quantitative signatures on the four primary elements. The dual-source system would employ a Cf-252 source for thermal neutron, prompt gammas and activation reactions, and a Pu-Be source for fast neutron, prompt gamma, and activation reactions.

#### *Neutron/Gamma Prototype Instrument*

The prototype neutron/gamma instrument is based on the results obtained from the feasibility study and consists of a thermal neutron cell (TNC), a fast neutron cell (FNC), an activation counting cell (ACC), and a control unit.

The TNC unit (Figure 1) contains a Cf-252 source (154  $\mu$ g on 15 August 1975) mounted on an 18-in. (45.7-cm) diameter rotating wheel, a motor and clutch system for driving the wheel, a 5 x 5 in. (12.7 x 12.7 cm) NaI(Tl) detector and preamp, and a junction box for interfacing to the control unit. The system is shielded in a 32-in. (81.3-cm) diameter steel cylinder filled with a borated-water extended polyester resin (WEP). Source-detector shielding consists of lead, tungsten, and lithium hydride. There is a 1 3/4-in. (4.4-cm) polymethyl methacrylate moderator between the source and the sample. Table 3 and Figure 2 provide the salient properties of the Cf-252 source.

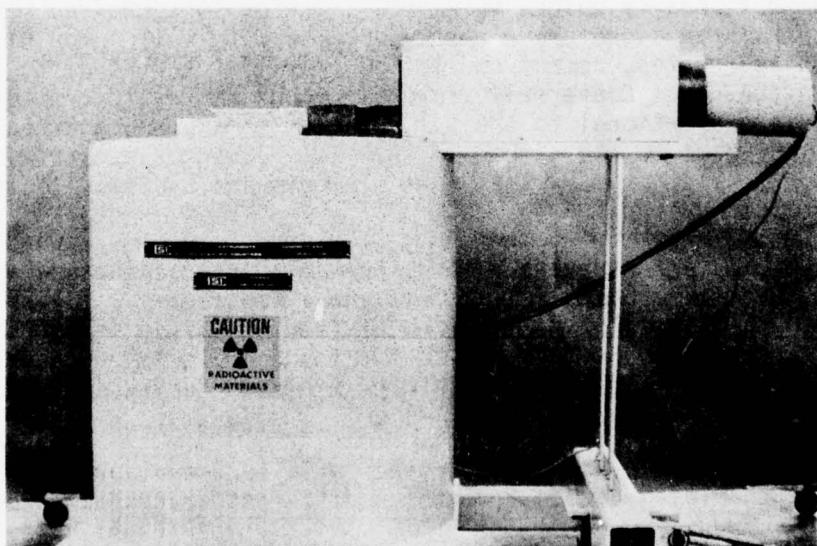


Figure 1. Neutron/gamma equipment, thermal neutron cell (TNC).

Table 2

## Feasibility Study Summary of Neutron/Gamma Methods for Concrete Analysis

Element	Importance	Technique	Reaction	Source	Detector	Gamma Energy (MeV)	Comments
Hydrogen	Primary	Thermal neutron capture	$H(n,\gamma)D$	$^{252}Cf$	NaI(Tl)	2.22	Best method.
					Ge(Li)	2.22	Better resolution, but less sensitive and more expensive.
		Fast neutron capture	$H(n,\gamma)D$	Pu-Be	NaI(Tl)	2.22	Less sensitive than thermal capture.
				14 MeV	NaI(Tl)	2.22	Least sensitive method.
Carbon	Primary	Thermal neut. capt.	$^{12}C(n,\gamma)^{13}C$	$^{252}Cf$	Ge(Li)	4.95	Very low intensity.
				$^{252}Cf$	NaI(Tl)	4.43	Very low intensity due to source spectrum.
		Neutron inelastic scattering	$^{12}C(n,n')^{12}C$	Pu-Be	NaI(Tl)	4.43	Best method provided source is well shielded.
					Ge(Li)	4.43	Offers no advantage due to Doppler broadening of gamma peak.
Silicon	Primary	Thermal neutron capture	$^{28}Si(n,\gamma)^{29}Si$	14 MeV	NaI(Tl)	4.43	Interference from $^{160}(n,n'\alpha)^{156}C$ for n energies > 12.5 MeV.
				$^{252}Cf$	Ge(Li)	3.53	Requires large expensive source.
		Neutron activation	$^{28}Si(n,p)^{28}Al$	$^{252}Cf$	NaI(Tl)	1.78	Works well with source unmoderated.
				Pu-Be	NaI(Tl)	1.78	Works very well - ~ 0.1% precision.
		Neutron inelastic scattering	$^{28}Si(n,n')^{28}Si$	14 MeV	NaI(Tl)	1.78	Works very well - ~ 0.1% precision.
				Pu-Be	NaI(Tl)	1.78	Works reasonably well.
				14 MeV	NaI(Tl)	1.78	Works reasonably well.

Table 2 (Cont'd)

Element	Importance	Technique	Reaction	Source	Detector	Gamma Energy (MeV)	Comments
Calcium	Primary	Thermal neutron capture	$^{40}\text{Ca}(n,\gamma)^{41}\text{Ca}$	$^{252}\text{Cf}$	Ge(Li)	6.42	Requires large expensive source.
		Neutron activation	$^{48}\text{Ca}(n,\gamma)^{49}\text{Ca}$	$^{252}\text{Cf}$	NaI (Tl)	3.09	Works well.
Aluminum	Secondary	Activation analysis	$^{27}\text{Al}(n,\gamma)^{28}\text{Al}$	$^{252}\text{Cf}$	NaI(Tl)	1.78	Requires well moderated source.
			$^{27}\text{Al}(n,p)^{27}\text{Mg}$	Pu-Be or NaI(Tl) 14 MeV	NaI(Tl)	.84	Can have interference from iron.
Magnesium	Secondary	Activation analysis	$^{24}\text{Mg}(n,p)^{24}\text{Na}$	Pu-Be or NaI(Tl) 14 MeV	NaI(Tl)	2.75	Works reasonably well.
Iron	Secondary	Activation analysis	$^{56}\text{Fe}(n,p)^{56}\text{Mn}$	Pu-Be or NaI(Tl) 14 MeV	NaI(Tl)	0.85	May require delay before count to avoid aluminum interference.



Table 3  
Nuclear Properties of  $^{252}\text{Cf}$

Mode of Decay	
Alpha Emission	96.9%
Spontaneous Fission	3.1%
Half-Life	
Alpha Decay	$2.731 \pm 0.007$ yr
Spontaneous Fission	$85.5 \pm 0.5$ yr
Effective (a and SF)	$2.646 \pm 0.004$ yr
Neutron Emission Rate	$2.31 \times 10^{12}$ N/(Sec-g)
Neutrons Emitted Per Spontaneous Fission	3.76
Average Neutron Energy	2.348 MeV
Average Alpha Particle Energy	6.117 MeV
Gamma Emission Rate (Exclusive of Internal Conversion X-rays)	$1.3 \times 10^{13}$ photms/sec-gr
Dose Rate at One Meter in Air	
Neutron	$2.2 \times 10^3$ rem/(hr-gr)
Gamma	$1.6 \times 10^2$ rads/hr-gr)
Decay Heat	
From Alpha Decay	18.8 w/g
From Fission	19.7 w/g
Source Volume (Excluding Void Space for Helium)	$<1 \text{ cm}^3/\text{g}$

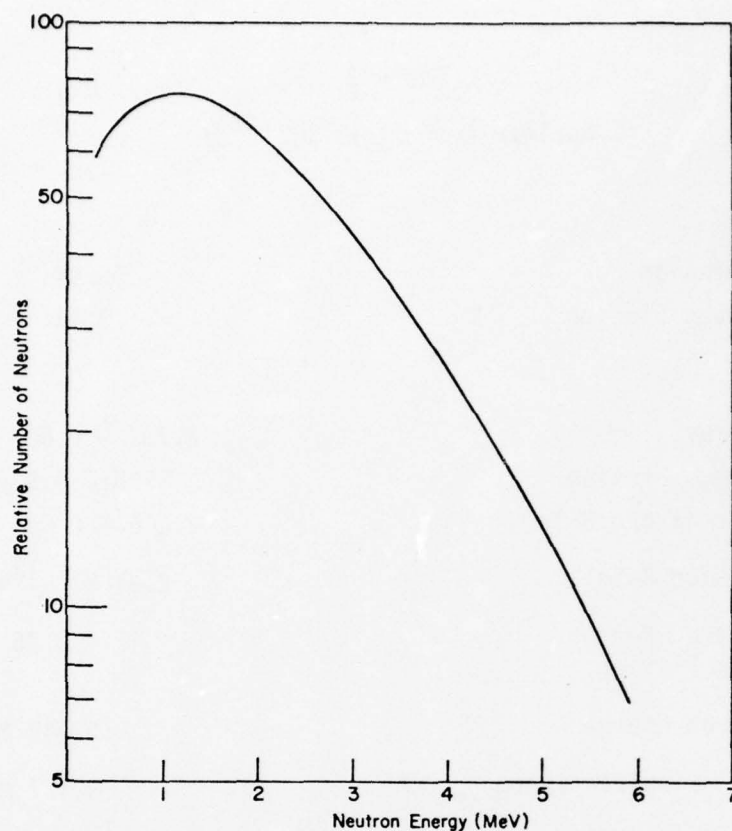


Figure 2. Neutron energy spectrum of  $^{252}\text{Cf}$ .

The FNC unit (Figure 3) contains an unmoderated 34-curie  $^{238}\text{Pu-Be}$  source mounted on a 15-in. (38.1-cm) diameter rotating wheel, housed in a WEP-filled 24-in. (60.9-cm) diameter steel cylinder. All other components of the FNC unit are identical to those of the TNC unit (motor, clutch, NaI(Tl) detector, etc.). Table 4 and Figure 4 provide the salient properties of the Pu-Be source.

The ACC unit (Figure 5) consists of a 5 x 5 in. (12.7 x 12.7 cm) NaI(Tl) detector, preamp, and junction box for interfacing to the control unit. The detector is shielded by lead rings in a 24-in. (60.9-cm) diameter, WEP-filled steel cylinder.

The electronic control unit (Figure 6) consists of a 1024 multi-channel analyzer, a high-voltage power supply, three linear amplifiers, a mixer-router module, a control module, and a powered nuclear instrumentation module (NIM) bin.

#### *Operating Procedures*

The equipment is operated by transmitting signals from each of the three detector systems to one of three amplifiers in the control

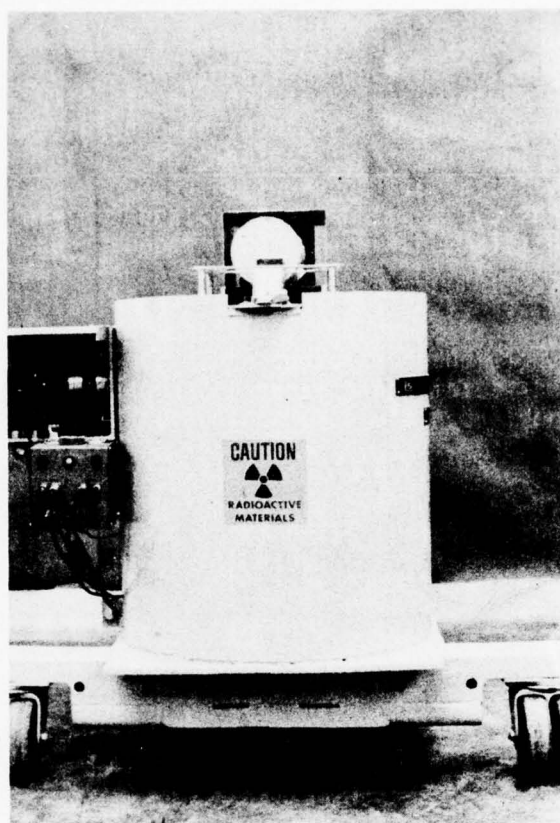


Figure 3. Neutron/gamma equipment fast neutron cell (FNC).

unit. The output pulses are then sent to the mixer/router module, which directs them to a specific 256-channel memory group (one of four available) in the multichannel analyzer. Each input from the mixer/router has a corresponding gate input from the control modules; thus, the analyzer accumulates a particular signal only when its gate is open. Each gate is open for a period specified by its present-time on the control module. Signal or event intensities are proportional to the area under the corresponding peak in the multichannel analyzer spectrum.

Figure 7, a flow diagram of the test procedure, illustrates the sequencing and present-times of the units and the signature elements analyzed by each. The standard test sequence is:

Table 4  
Nuclear Properties of  $^{238}\text{Pu}$ -Be Source

Mode of Decay $^{238}\text{Pu}$	
Alpha Emission	>99.999%
Spontaneous Fission	<.001%
Half Life $^{238}\text{Pu}$	
Alpha Decay	86.4 yr
Spontaneous Fission	$4.9 \times 10^{10}$ yr
Effective (X + sq ft)	86.4 yr
Alpha - Neutron Target Reaction	${}^9_4\text{B} + {}^4_2\text{He} \rightarrow {}^{12}_6\text{C} + {}^1_0\text{n} + \text{Energy}$
Neutron Emission Rate (approx)	$2.2 \times 10^6$ (n/Sec-Ci)
Average Neutron Energy (approx)	4 MeV
Alpha Particle Energies	5.50 (72%), 5.46 (28%) MeV
Dose Rate at 1 m in Air	
Neutron	2.5 M rem/hr-Ci
Gamma	0.01 M rad/hr-Ci
Decay Heat	0.0242 w/Ci
Source Volume (Internal Requirements)	$0.5 \text{ cm}^3/\text{Ci}$

1. Fill two constant-volume stainless-steel containers (8 in. [20.4 cm] diameter x 5 in. [12.7 cm] high) with the material (concrete) to be analyzed. Determine the weight of material in the containers.

2. Place one sample container on the TNC unit and the other on the FNC unit. Start irradiation and counting (5 min for the TNC unit, and 10 min for the FNC unit).

3. After completing the 5 min of TNC irradiation and counting, transfer the TNC sample to the activation counting cell (1 min is allowed for the transfer).



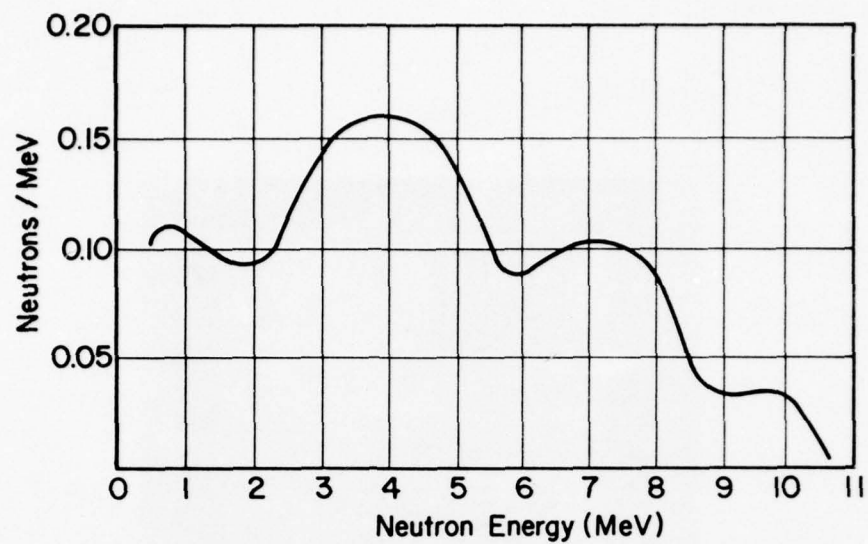


Figure 4. A typical  $(\alpha, n)$  source spectrum for Pu-Be. (From B. T. Price, C. C. Horton, and K. T. Spinney, *Radiation Shield* [Pergamon Press, 1957], p 151.)

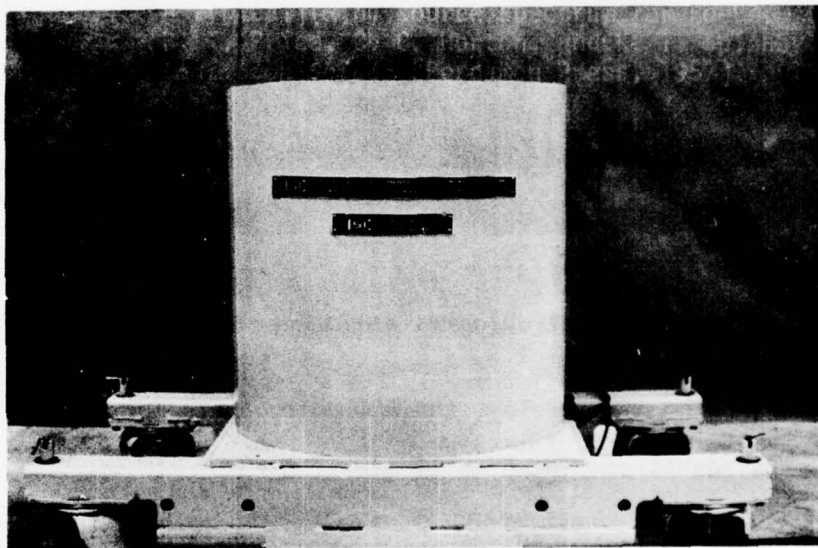


Figure 5. Neutron/gamma equipment, activation counting cell (ACC).

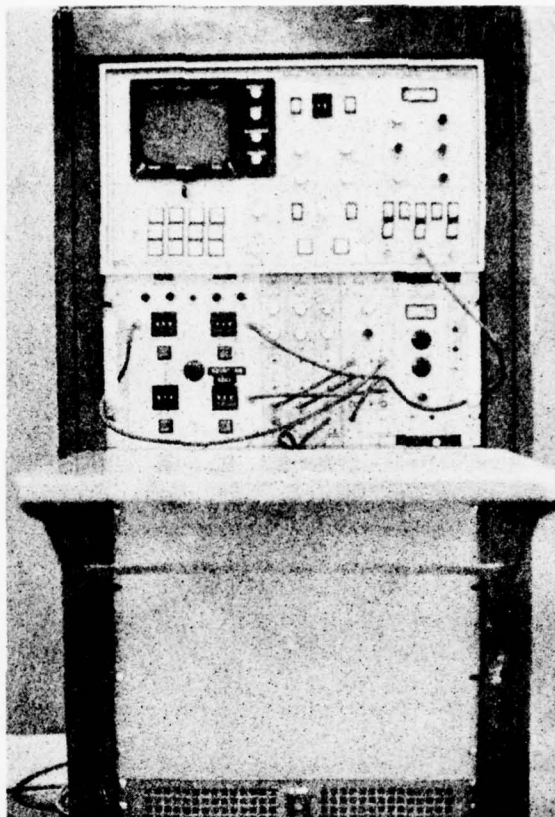


Figure 6. Neutron/gamma equipment, control unit.

4. After the 1-min delay, the ACC unit counts for 5 min.
5. The run is completed in 11 min.

After the run is completed, the analyzer's collect function is switched off, and the collected gamma spectra from each of the three detectors can be displayed on the scope for analysis. The analyzer also digitalizes the collected data on a per-channel, per-event basis. The unit can sum or integrate the intensities of any consecutive group of channels.

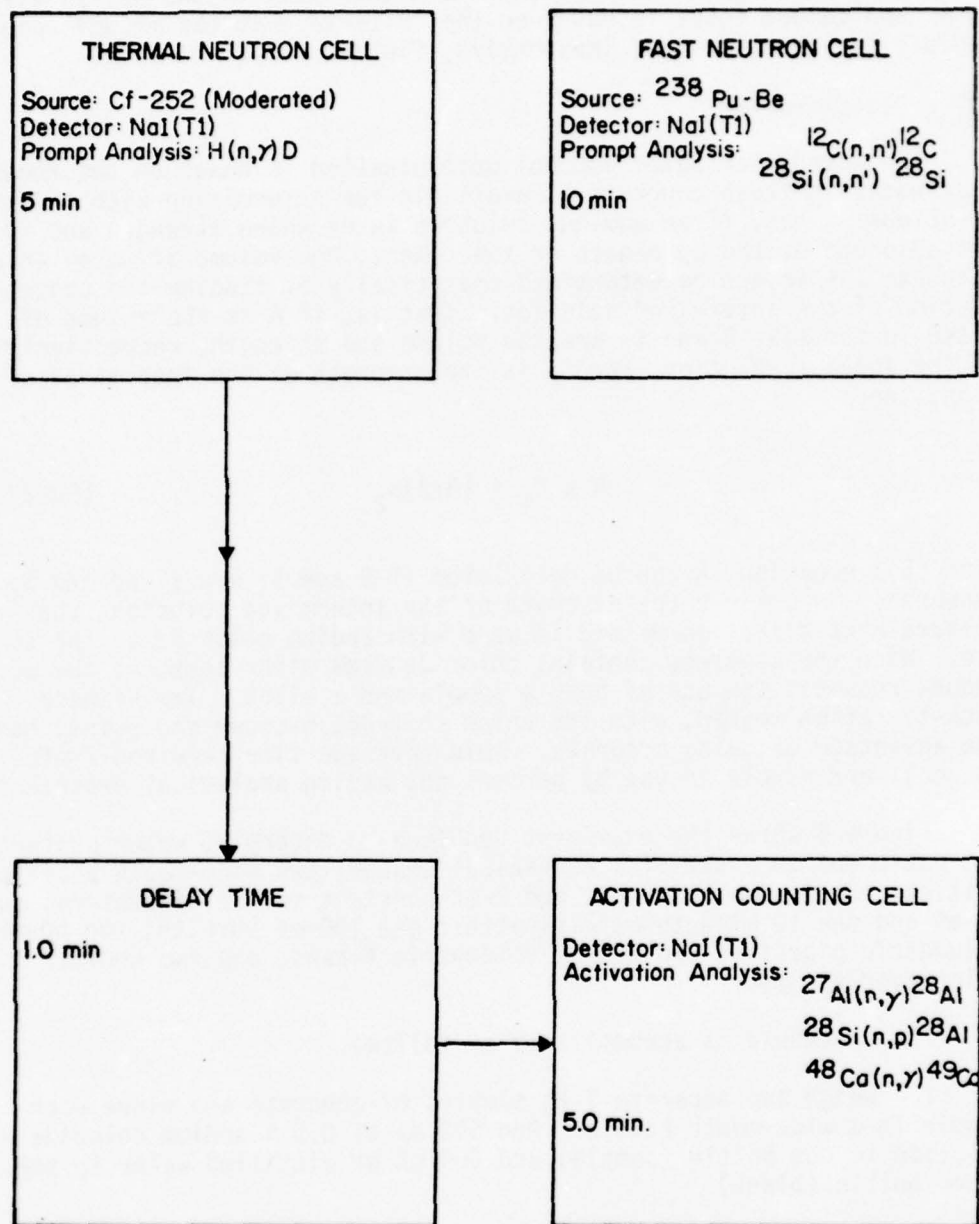


Figure 7. Analysis procedure flow diagram for neutron/gamma system.

### Kelly-Vail Technique

The selection of the chemical techniques used in the Kelly-Vail water and cement tests is based on the criteria that the methods are rapid (less than 15 min), inexpensive, field-worthy, and safe.

#### *Water Determination*

The method for water content determination is based on the theory that water in fresh concrete is available for intermixing with aqueous solutions. Thus, if an aqueous solution is of known strength and is not absorbed by the aggregate or the cement, the volume of water in a concrete sample can be determined analytically by finding the concentration of the intermixed solution. That is, if A is the volume of water in the mix, B and  $S_1$  are the volume and strength, respectively, of the aqueous solution, and  $S_2$  is the strength of the intermixed solution, then:

$$B \times S_1 = (A+B)S_2 \quad [\text{Eq 2}]$$

From this equation, A can be calculated if B and  $S_1$  are fixed and  $S_2$  is measured. To measure the strength of the intermixed solution, the Volhard back-titration method is used with sodium chloride as the solute. When the concrete contains chloride from other sources, the procedure requires the use of both a sample and a blank. The Volhard back-titration method, with its white to reddish-brown end point, has the advantage of being accurate, rapid (average time required 7 min, 30 sec), and simple to use by persons not having analytical experience.

Figure 8 shows the equipment required to determine water content. The equipment consists of a mechanical shaker; two wide-mouth plastic bottles; 10-ml, 5-ml, 2.5-ml, and 2-ml constant volume dispensers; two 50-ml and one 10-ml automatic pipettes; one 100-ml burette; two 50-ml volumetric pipettes; two 500-ml volumetric flasks; and two 500-ml Erlenmeyer flasks.

The procedure is accomplished as follows:

1. Weigh two separate 1-kg samples of concrete and place each sample in a wide-mouth bottle. Add 500 ml of 0.5 N sodium chloride solution to one bottle (sample) and 500 ml of distilled water to the other bottle (blank).
2. Seal the bottles and place them in a mechanical shaker for 3 min.
3. Pipette a 50-ml sample of clear supernatant liquid from the sample and blank bottles and add to separate Erlenmeyer flasks. To



each flask (sample and blank), add 10 ml of 50 percent nitric acid, 2 ml of nitrobenzene, and 5 ml of ferric alum. Shake well.

4. Determine the chloride content of the sample and blank flasks by adding excess silver nitrate (50 ml of 0.5 N  $\text{AgNO}_3$  for sample and 10 ml of 0.5 N  $\text{AgNO}_3$  for blank) and back-titrating with 0.05 N potassium thiocyanate (Volhard back-titration).

5. Record the quantity of potassium thiocyanate (KCNS) required to reach the white to reddish-brown end point in both the sample and the blank. Use Figure 9 to determine the water content of the mix.\*

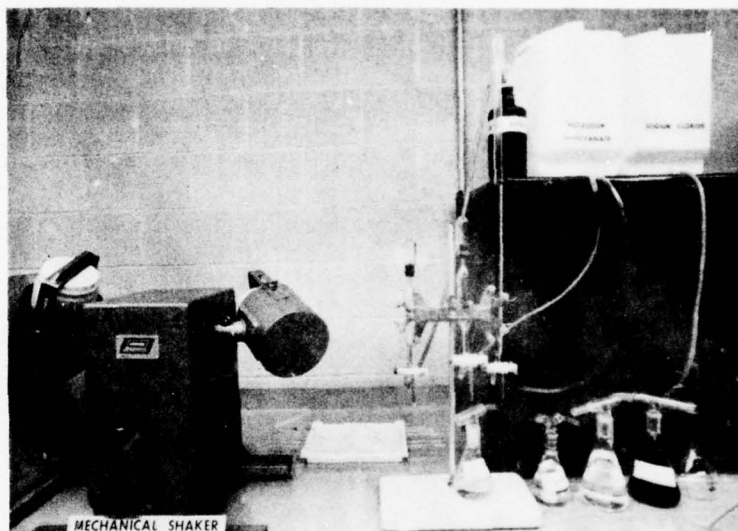


Figure 8. Equipment required for determination of water content by Kelly-Vail technique.

#### *Cement Determination*

The cement determination technique is based on the assumptions that: (1) cement can be dispersed in water and held uniformly in suspension so that a representative sample can be obtained; (2) a quantitative solution of the cement in nitric acid can be achieved by adding

\* Quantity of KCNS (ml) required for sample titration plus the back-titration of the blank (100 minus the KCNS required for blank titration) equals the abscissa of Figure 9.

cement to the acid while rapidly stirring without external heat; and (3) calcium can be determined by a flame photometer in relatively high concentrations in the nitric acid solutions without prior removal of silica and the sesquioxides.

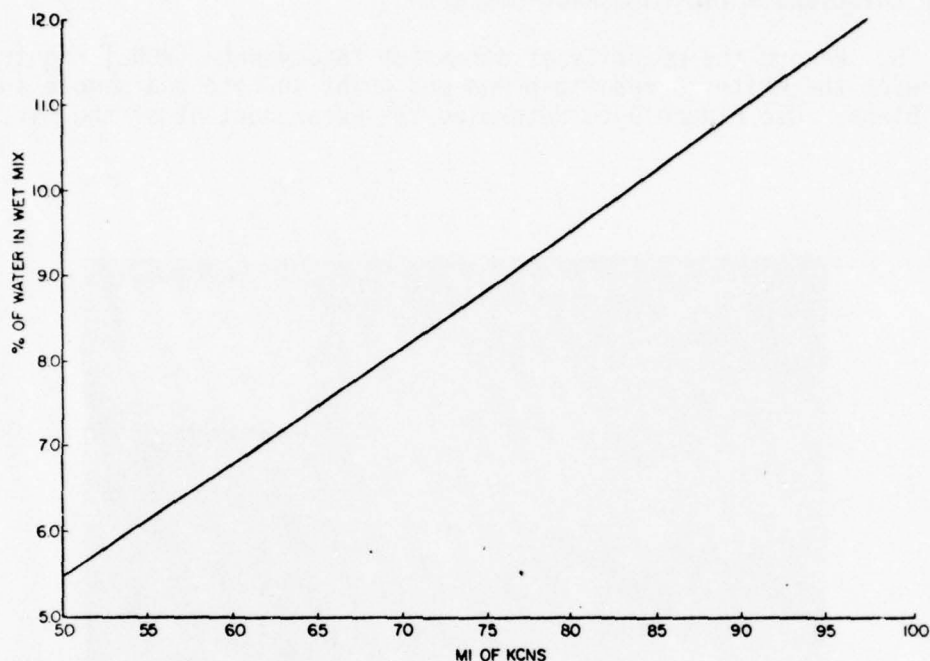


Figure 9. Relationship between water content and KCNS required for titration (Kelly-Vail).

Figure 10 shows the equipment required for the cement tests. The apparatus for preparing and sampling the cement-water suspension consists of a nest of sieves (No. 4 and No. 50) over a side-agitator domestic washing machine and three automatic pipettes. One pipette collects the constant volume cement-water sample from the washing machine; the others dilute the sample with nitric acid and water. An ordinary domestic high-speed stirrer (milkshake type) provides agitation for dissolving the cement suspended in the acid solution. A flame photometer determines the calcium (cement) concentration.

Briefly, the major steps are as follows:

1. Fill the washing machine with 10 gal (37.9 l) of tap water; place nest of sieves over the machine; start agitator and pump to recirculate water.

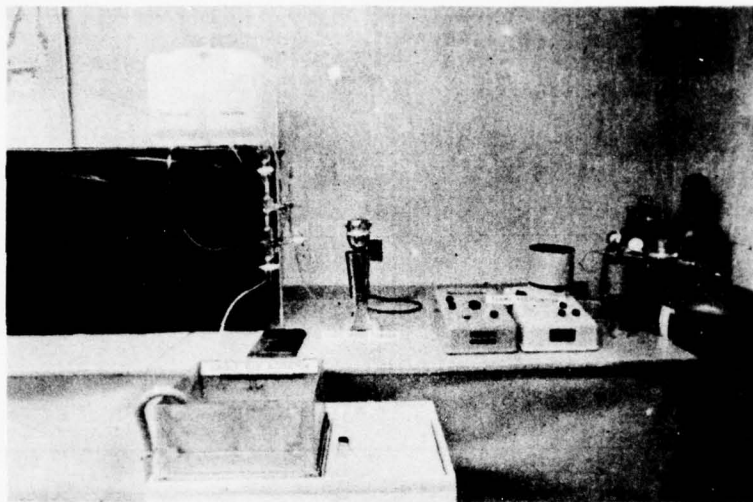


Figure 10. Equipment required for determination of cement content by Kelly-Vail technique.

2. Place a 1-kg concrete sample on the nest of sieves and wash the cement from aggregate particles with the recirculating hose.
3. Allow agitation-recirculation operation to continue for 3 min. Attach the small hose to the automatic pipettes; then clamp the recirculating hose nozzle so that the cement suspension will flow through the small hose and fill the automatic pipette (125 ml).
4. Empty the sample of cement suspension into a mixing cup and wash down the pipette with 100 ml of 5 percent nitric acid from the upper pipette. Concurrently, dilute the acid-cement solution with 300 ml of tap water from the third pipette.
5. Stir the contents of the mixing cup in the high-speed mixer for 3 min.
6. Calibrate the flame photometer with a calcium standard and measure the calcium content of solution in the mixing cup. See Figure 11 to convert the readout to cement content. (Calcium standard is equal to 240 g of cement added to 10 gal (37.9 l) of water in the washing machine, or approximately 0.94 g/l of  $\text{CaCO}_3$ ). The average time for a cement determination by an experienced operator is 7 min, 10 sec.

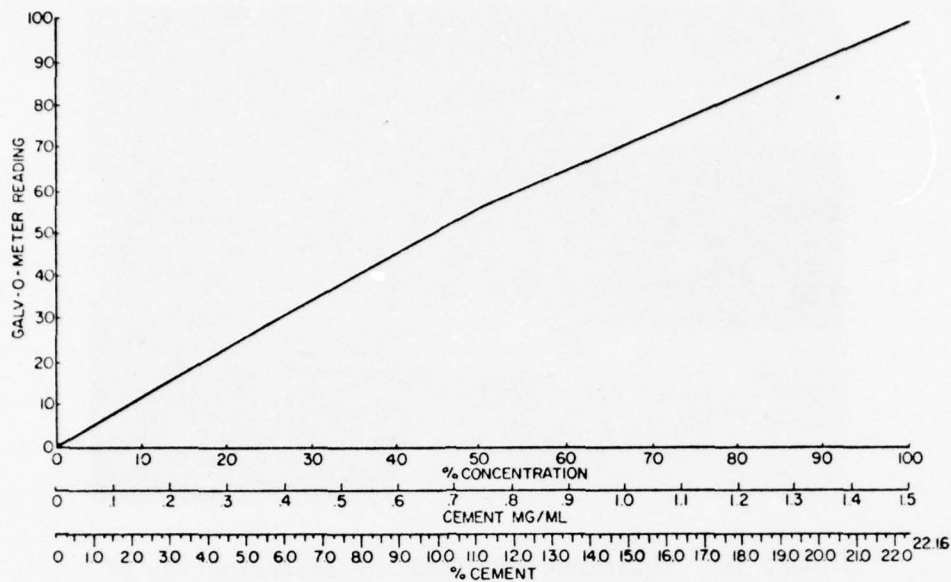


Figure 11. Relationship between Galv-o-meter reading and cement content (Kelly-Vail).



## 4 LABORATORY TESTS

### Mortar Test Series

#### *Test Materials, Mix Proportions, and Mix Procedure*

The initial test series ran neutron/gamma tests on a series of mortar mixes and their constituents.

The materials used in the mortar test series were a type I portland cement, tap water, ottawa sand, siliceous river sand, and a crushed limestone sand. To minimize the variations in the constituents' chemical compositions, all the cement used in the mortar test series was obtained at one time from one source and supplier. Similarly, each of the sands was obtained at one time from given suppliers.

The mortar constituent tests evaluated both oven dry and moist samples of the cement and of each sand. Tables 5, 6, and 7 list the mix proportions for the cement-water and sand-water mixes. Three mortar test series were evaluated--one for each of the three sands used. Each series evaluated three sand-cement ratios and three water contents for each sand-cement ratio (see Tables 8, 9, and 10 for mix proportions).

The mix procedure for both the constituent and mortar tests consisted of oven drying ( $105 \pm 5^\circ\text{C}$ ) the sand for 24 hours, weighing out the mix constituents so that each batch weighed approximately 60 lb (24 kg), and mixing the ingredients in a 1-cu ft ( $.02\text{-m}^3$ ) capacity laboratory bowl mixer until mix homogeneity was obtained (normally 3 to 5 min).

#### *Test Procedure*

The test procedure consisted of filling the two 8-in. (20.4-cm) diameter by 5-in. (12.7-cm) high stainless steel sample containers with the material to be tested. The containers were tamped as the material was added. To obtain a constant volume sample, the containers were filled completely and the tops evenly smoothed off. The net weight of each sample was determined to the nearest tenth of a pound. A standard neutron/gamma test was conducted on all samples (mortar, cement paste, and sand-water mixes). For details of the neutron/gamma operating procedure, see Chapter 3.

After completion of the irradiation-counting cycle for each neutron/gamma test, the TNC spectrum was analyzed for the 2.22 MeV prompt gamma rays from the  $\text{H}(\text{n}, \gamma) \text{H}$  neutron capture reaction. The FNC spectrum was analyzed for the 4.43 and the 1.78 MeV prompt gamma

Table 5  
Mortar Test Series Constituent Test: Cement/Paste Elemental Signature Intensities

Mix Proportions (%)		Sample Weights (lbs)		Elemental Signature Intensities					
		Cement	TNC-ACC	FNC	H (TNC)	Si (FNC)	C (FNC)	Al-Si (ACC)	Ca (ACC)
Water									
0.0		100.0	12.8	12.5	1920	129794	12505	76087	4714
2.1		97.9	12.4	12.3	7696	126420	10531	90353	5558
3.8	(1)	96.2	11.9	11.8	11143	109879	10066	94794	5794
7.4		92.6	11.9	12.1	21286	109589	11364	108931	6979
10.7		89.3	12.2	12.6	30017	113383	12003	122492	8005
13.8		86.2	18.8	17.3	64630	102643	10856	214353	14954
16.7	(1)	83.3	20.4	20.6	75066	98600	10993	209511	14885
19.4		80.6	20.3	20.2	84360	95990	13307	194666	14528
21.9		78.1	20.0	20.0	101695	94726	14106	219328	15138
24.3	(2)	75.6	19.1	19.1	122007	94356	13992	219049	15299
26.5	(1,2)	73.5	18.4	18.5	138505	110316	10346	218167	15519
28.6	(1,2)	71.4	17.8	18.1	147383	93698	13501	208555	15150

Notes: (1) First run of day  
(2) Bleeding

Table 6

Mortar Test Series Constituent Test: Ottawa Sand/Water Element Signature Intensities

Mix Proportions (%)		Sample Weights (lbs)		Elemental Signature Intensities					
		Sand	TNC-ACC	FNC	H (TNC)	Si (FNC)	C (FNC)	Al-Si (ACC)	Ca (ACC)
Water									
0.0	100.0		14.9	14.7	3807	400617	14670	80467	(1)
2.1	97.1		13.9	13.2	10209	380972	14869	81807	(1)
3.8 (2)	96.2		15.2	15.3	16033	374119	11846	65825	(1)
7.4	92.6		15.4	15.9	27989	349859	12395	72487	(1)
10.7	89.3		16.0	16.2	40742	338136	14210	70474	(1)
13.8	86.2		17.0	17.4	65624	319708	15046	75655	(1)
16.7 (2)	83.3		17.8	17.6	83515	305902	13481	62101	(1)
19.4	80.6		18.0	17.7	96210	297660	14618	71541	(1)

\*Note: (1) No detectable Ca peak.

(2) First run of day.

Table 7

## Mortar Test Series Constituent Test: Elemental Signature Intensities

Mix Proportions (%)		Sample Weights (lbs)		Elemental Signature Intensities									
				Cement	TNC-ACC		FNC	H (TNC)	Si (FNC)	C (FNC)	Al-Si (ACC)	Ca (ACC)	
Water													
0.9 (1)	100.0	13.7	13.8	1764	(2)	10507	94447	5624					
3.8	96.2	12.2	12.2	10591	77424	14553	106510	6344					
10.7	89.3	13.2	13.3	36581	67837	14611	151441	9421					
16.7	83.3	20.6	20.5	79006	76394	13678	249234	16550					
21.9 (1)	78.1	20.1	20.1	105974	62897	15741	234059	15926					
River Sand													
Water													
0.9	100.0	16.0	14.8	5733	262322	20369	156932	1803					
3.8	96.2	15.8	15.7	19527	257661	22030	177882	2026					
10.7	89.3	17.0	17.3	55441	210581	19700	230208	2673					
16.7	83.3	18.2	17.8	80176	203589	20536	227021	2264					
Limestone Sand													
Water													
0.9 (1)	100.0	16.7	16.9	3328	(2)	33097	35008	6402					
3.8	96.2	16.2	16.4	13210	10610	33219	47475	8330					
10.7	89.3	20.6	20.6	49605	34550	33006	70573	13885					

\*Note: (1) First run of day.

(2) Si background channels were below the lower threshold of the data processed by the Multichannel Analyzer.



Table 8  
Mortar Test Series Mortar Test: Ottawa Sand/Mortar Elemental Signature Intensities

Mix Proportions (%)			Sample Weights (lbs)		Elemental Signature Intensities					
Water	Cement	Sand	TMC-ACC	FNC	H (TNC)	Si (FNC)	C (FNC)	Al-Si (ACC)	Ca (ACC)	
7.9	23.0	69.0	19.2	19.0	31813	297083	13048	111160	4255	
9.1 (1)	22.7	68.1	19.7	19.8	40534	288166	9771	113380	4182	
10.4	22.4	67.2	20.1	20.0	48558	280124	13403	116063	4561	
7.9	18.4	73.7	19.3	19.0	33538	311849	14585	107857	3723	
9.1	18.2	72.7	19.6	19.6	40172	305840	12636	110412	3753	
10.4 (1)	17.9	71.7	19.8	19.14	53193	308617	10318	105657	3654	
7.9	13.2	79.0	17.9	18.3	34192	321063	14139	88328	2494	
9.1	13.0	77.9	18.7	17.8	40769	313921	13632	98182	3057	
10.4	12.8	76.8	(2)	(2)	(2)	(2)	(2)	(2)	(2)	

Notes: (1) First run of day.  
(2) Equipment failure; test not completed.

Table 9  
Mortar Test Series Mortar Test: River Sand/Mortar Elemental Signature Intensities

Mix Proportions (%)			Elemental Signature Intensities					
			Sample Weights (lbs)		H	Si	C	Ca
Water	Cement	Sand	TNC-ACC	FNC	(TNC)	(FNC)	(FNC)	(ACC)
7.9	23.0	69.0	19.2	18.9	35695	182181	16533	229079
9.1	22.7	68.1	20.6	20.6	43515	180825	16228	245368
10.4	22.4	67.2	20.2	20.2	48091	192522	19601	245660
7.9	18.4	73.7	19.2	19.2	34903	201795	18156	240715
9.1 (1)	18.2	72.7	20.2	20.2	41341	185946	17150	233708
10.4	17.9	71.7	20.0	20.0	46516	192689	17888	240580
7.9 (1)	13.2	79.0	19.0	19.2	37324	204987	15919	214458
9.1	13.0	77.9	19.5	19.6	41982	204580	17271	233318
10.4	12.8	76.8	19.6	19.6	48973	205033	18366	243284
								4573

Notes: (1) First run of day.

Table 10

Mortar Test Series Mortar Test: Limestone Sand/Mortar Elemental Signature Intensities

Mix Proportions (%)			Sample Weights (lbs)		Elemental Signature Intensities					
Water	Cement	Sand	TNC-ACC	FNC	H (TNC)	Si (FNC)	C (FNC)	Al-Si (ACC)	Ca (ACC)	
7.9	23.0	69.0	20.4	20.4	35159	32566	27920	118030	13834	
9.1 (1)	22.7	68.1	22.0	22.0	42454	(2)	24123	113546	14688	
10.4	22.4	67.2	21.4	21.4	46986	41675	27992	113500	14639	
7.9	18.4	73.7	21.2	21.1	34509	34113	28155	111157	14624	
9.1 (1)	18.2	72.7	21.6	21.8	42142	(2)	24510	110045	13881	
10.4	17.9	71.7	21.0	21.3	49647	(2)	27885	113391	14701	
7.9	13.2	79.0	21.4	21.6	36633	27220	27449	102505	14305	
9.1	13.0	77.9	21.7	21.7	43331	34126	27234	112684	14155	
10.4	12.8	76.8	21.2	21.3	49707	(2)	27183	86254	13483	

Notes: (1) First run of day.

(2) Si background channels were below the lower threshold of data processed by the Multichannel Analyzer  
Thus, net Si peak was invalid.

rays from, respectively, the  $^{12}\text{C}(\text{n}, \text{n}')^{12}\text{C}$  and the  $^{28}\text{Si}(\text{n}, \text{n}')^{28}\text{Si}$  neutron inelastic scattering reactions. The spectrum from the ACC was analyzed for the 3.09 MeV decay gamma rays from the  $^{48}\text{Ca}(\text{n}, \gamma)^{49}\text{Ca}$  neutron activation reaction, and for the 1.78 MeV decay gamma rays from both the  $^{27}\text{Al}(\text{n}, \gamma)^{28}\text{Al}$  and the  $^{28}\text{Si}(\text{n}, \text{p})^{28}\text{Al}$  neutron activation reactions.

Spectral analysis involved determining the net peak intensities for the above five gamma energies by subtracting background intensities from peak intensities. Peak intensities were taken as bands six channels wide for the 1.78 MeV  $^{28}\text{Si}$  peak FNC spectrum, and eight channels wide for the 4.43 MeV  $^{12}\text{C}$  peak FNC spectrum and for the 1.78 MeV  $^{28}\text{Al}$  and the 3.09 MeV  $^{49}\text{Ca}$  peaks ACC spectra. Background intensities were computed by skipping one channel and counting half the number of peak channels on both sides of the peak bands.

The report on the neutron/gamma system indicated that the background channels for the TNC 2.22 MeV six-channel H peaks were overriding a 1.94 MeV Ca peak, thus allowing the Ca content of the sample to interfere with the background count of the H signature.<sup>10</sup> To evaluate this problem, individual channels were counted for the 10 channels on both sides of the H peak. This recording technique was used on the ottawa and mortar series, and the cement paste and ottawa sand-water constituent tests. The river sand and limestone sand mortar and constituent tests used a four-channel peak band with two-channel background.

Due to the slight drift in the location of the peak channels, net peak intensities were computed for an entire series of band locations. This continued until the maximum net peaks and net peaks for bands starting one channel on each side had been obtained. This insured that the maximum net peak was always obtained. Only the maximum net peak intensities were used in the data analysis.

#### *Test Results*

Tables 5 and 6 list the signature (net peak) intensities obtained for the cement paste and ottawa sand-water constituent tests. Table 8 gives the signature intensities obtained for the ottawa sand mortar mixes. The H signatures listed in Tables 5, 6, and 8 are based on four-channel peaks and two 2-channel backgrounds.

The last ottawa sand-mortar test was not completed due to an equipment failure in the photomultiplier component of the NaI detector on the FNC. It was decided that the cement paste constituent tests

<sup>10</sup> P. A. Howdysshell, "Preliminary Evaluation of the Neutron/Gamma Technique to Determine the Water and Cement Content of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (CERL, May 1975).



should be partially rerun after the photomultiplier was repaired (proper electrical grounding). Table 7 provides these results and those of the river sand-water and limestone sand-water constituent tests. The test results of the river sand and limestone sand mortars are listed in Tables 9 and 10, respectively.

### Concrete Test Series

#### *Test Materials, Mix Proportions, and Mix Procedure*

The concrete tests ran neutron/gamma and Kelly-Vail tests on four concrete test series and neutron/gamma and Kelly-Vail cement content tests on each material constituent used in the concrete tests.

The materials used in the four test series were a type I portland cement, tap water, siliceous river sand, crushed limestone sand, 3/4-in. (1.90-cm) gravel, and a 3/4-in. (1.90-cm) crushed limestone. All materials required to complete the tests were ordered and stockpiled before the four test series began. A chemical analysis on all five material constituents was conducted to determine their Si, Ca, Al, Fe, Mg, and C contents (see Table 11). Table 12 lists the aggregate gradations.

Table 11  
Chemical Analysis - Concrete Constituents

Constituent	Si %	Fe %	Al %	Mg %	Ca %	C %
Cement	10.0	1.8	4.0	1.2	46.2	0
River Sand	31.5	0.6	6.7	3.1	5.3	3.1
Limestone Sand	6.8	1.2	9.6	2.0	28.0	9.4
3/4-in Gravel	19.0	1.2	6.8	4.4	11.5	5.6
3/4-in Limestone	3.5	0.6	0.5	0.5	35.6	10.9

Notes: Gravimetric analysis was used. Results were based on two samples per constituent.

Table 12  
Sieve Analysis--Concrete Aggregates

Sieve Size	River Sand	Percent Passing Limestone Sand	3/4 Gravel	3/4 Limestone
1-in			99.9	100
3/4-in			99.5	94.0
1/2-in			91.5	53.6
3/8-in	100	100	71.9	25.6
No 4	98.7	100	11.7	2.1
No 8	89.8	87.8	6.7	1.1
No 16	78.8	62.1	6.0	0.9
No 30	60.8	32.1	5.5	0.8
No 50	22.6	15.0	4.8	0.7
No 100	1.2	4.4	3.2	0.6
No 200	0.5	1.6	2.0	0.5
Pan	0	0	0	0

Neutron/gamma tests were conducted on both oven-dry and moist samples of each of the five material constituents. Tables 13, 14, and 15 list the mix proportions for the constituent-water tests. The four concrete test series consisted of cement having (1) river sand and 3/4-in. (1.90-cm) gravel; (2) river sand and 3/4-in. (1.90-cm) limestone; (3) limestone sand and 3/4-in. (1.90-cm) limestone; and (4) limestone sand and 3/4-in. (1.90-cm) gravel. Table 16 gives the mix proportions of the four concrete test series.

For mixing, the constituents were oven-dried at  $105 \pm 5^\circ\text{C}$  for 24 hours. After the materials had cooled to room temperature, samples were either tested immediately or mixed with water and tested. The mixing procedure and equipment for the cement and sand-water tests were the same as those used for the mortar tests and their constituents. For the moist, coarse-aggregate constituents, the materials were immersed in water for 24 hours and then tested in an immersed state or surface dried to obtain saturated surface dry samples and tested.

Table 13

Concrete Test Series Constituent Test: Cement/Water Elemental Signature Intensities

Mix Proportions		Sample Weight		E L E M E N T A L   S I G N A T U R E   I N T E N S I T I E S											
(%)		(lbs)		H (TNC)		Si (FNC)		C (FNC)		Al-Si (ACC)		Ca (ACC)			
Water	Cement	TNC-ACC	FNC	Net	Net/PHA T	Net	Net/PHA T	Net	Net/PHA T	Net	Net/PHA T	Net	Net/PHA T		
0.0	100.0	13.5	13.6	3887	16.90	123189	258.8	5269	11.07	101453	399.4	5368	21.10		
0.0	100.0	13.7	13.6	5079	22.18	131109	277.2	8228	17.40	96714	382.3	5408	21.38		
0.0	100.0	12.6	13.0	4927	21.61	125842	266.6	6964	14.75	95867	378.9	5287	20.90		
0.0	100.0	12.6	13.0	4758	20.69	131075	276.5	7372	15.55	96638	380.5	5397	21.25		
0.0	100.0	12.2	12.5	4700	20.43	122781	257.94	6378	13.40	81786	319.5	4745	18.45		
3.85	96.15	12.1	12.2	15467	66.68	114906	240.39	5523	11.55	113271	444.2	6425	25.20		
3.85	96.15	11.5	11.7	14139	61.74	122562	258.6	5762	12.16	94514	370.6	5413	21.23		
10.7	89.3	13.5	13.8	40785	178.88	102084	215.8	5223	11.04	147914	582.3	9089	35.78		
10.7	89.3	12.8	13.0	40298	178.30	112930	240.8	7225	15.40	133464	527.5	8128	32.13		
13.8	86.2	18.5	16.6	83077	370.88	104634	225.99	8167	17.64	223235	900.14	13890	56.01		
16.7	83.3	20.4	19.5	102055	466.31	93594	203.91	6696	14.59	256164	1041.32	16054	65.26		
16.7	83.3	20.4	20.4	95226	432.84	96670	212.46	8641	18.99	232242	947.93	14662	59.64		
19.4	80.6	20.5	20.1	112873	513.06	93788	205.68	8452	18.54	239607	977.99	14720	60.08		
21.9	78.1	20.2	20.1	133119	610.64	87562	186.30	8377	17.82	252504	1026.44	15721	63.91		
21.9	78.1	19.9	20.1	129283	590.33	90143	198.12	10191	22.40	224843	910.30	14736	59.66		
24.3	75.7	19.4	19.0	155769	711.27	91082	201.06	11157	24.63	246460	1005.96	15476	63.17		
26.5	73.5	18.4	19.0	162650	753.01	90347	200.32	10887	24.14	231659	949.42	15330	62.83		

Table 14

Concrete Test Series Constituent Test: Fine Aggregate/Water Elemental Signature Intensities

Mix Proportions %		Sample Weight (lbs)	Elemental Signature Intensities									
			H (TNC)		SI (FNC)		C (FNC)		Al-Si (ACC)		Ca (ACC)	
			Net	Net/PHAT	Net	Net/PHAT	Net	Net/PHAT	Net	Net/PHAT	Net	Net/PHAT
Water	River Sand	TNC-ALL	FNC									
0	100	15.2	15.4	20.24	32300	687.23	13545	28.82	136479	543.74	1607	6.40
0	100	15.1	15.6	19.87	319044	678.82	13653	29.05	133198	528.56	1200	4.76
0	100	15.2	15.2	19.97	314154	669.84	14873	31.71	128235	508.87	1082	4.29
0	100	15.7	15.5	23.59	299824	635.22	11834	25.07	136000	537.55	995	3.93
3.85	96.15	15.4	15.4	84.23	285625	607.71	12350	26.28	170720	674.78	1756	6.94
10.7	89.3	16.8	16.7	254.07	252744	543.54	12957	27.86	225054	900.22	2694	10.78
16.7	83.3	18.0	17.5	397.78	234443	505.26	11949	25.75	224007	892.46	2258	9.00
Water	Limestone Sand											
0	100	15.4	16.0	23.74	113399	240.25	21746	46.07	116690	464.06	4811	19.09
0	100	15.3	15.8	27.76	113285	239.50	22117	46.76	107241	423.88	4474	17.68
0	100	15.2	15.7	23.10	115261	243.68	21945	46.40	100251	394.69	4222	16.62
0	100	15.2	15.4	32.25	107477	227.22	20646	43.65	96189	378.70	3910	15.39
2.2	97.8	15.1	15.5	93.71	91265	192.54	17845	37.65	124468	490.03	5421	21.34
10.7	89.3	17.2	17.3	264.26	91095	194.65	18208	38.90	195534	777.12	8738	34.67
16.7	83.3	19.7	19.9	450.33	87993	190.46	17767	38.46	229663	918.54	10497	41.99



Table 15

Concrete Test Series Constituent Test: Coarse Aggregates/Water Elemental Signature Intensities

Mix Proportions (%)		Sample Weight (lbs)		E L E M E N T A L   S I G N A T U R E   I N T E N S I T I E S									
				H (FNC)		Si (FNC)		C (FNC)		Al-Si (ACC)		Ca (ACC)	
				Net	Net/PHA T	Net	Net/PHA T	Net	Net/PHA T	Net	Net/PHA T	Net	Net/PHA T
Water	3/4-in. Gravel	TNC-ACC	FNC										
0.0	100.0	14.2	14.2	5981	26.00	217620	460.08	16106	34.05	113768	447.90	1833	7.22
0.0	100.0	14.2	14.4	5098	22.16	2.9452	463.96	16869	35.66	112914	444.54	2196	8.64
0.0	100.0	14.3	14.2	4529	19.78	219639	466.32	16125	34.24	108461	430.40	1755	6.96
0.0	100.0	15.0	14.4	6010	26.24	216636	463.21	16907	35.82	130221	516.75	2079	8.25
2.85	97.15	16.1	15.7	14284	62.65	211252	447.58	16395	34.74	140521	553.23	2525	9.94
22.5	77.5	17.8	18.1	161812	728.88	156707	339.93	16062	34.84	211836	850.75	4745	19.06
22.1	77.9	18.8	19.0	153770	695.79	161761	347.13	15938	34.20	206970	808.48	4447	17.37
Water	3/4-in. Limestone												
0.0	100.0	12.9	13.2	3725	16.06	64048	133.43	25388	52.89	22675	87.89	4660	18.06
0.0	100.0	13.4	13.2	3661	15.85	62112	129.94	24300	50.84	17882	69.58	4178	16.26
0.0	100.0	12.7	12.9	3592	15.55	67354	141.20	24302	50.95	22478	87.46	4254	16.55
0.0	100.0	13.6	14.1	3559	15.41	70521	147.84	21789	45.68	40052	157.07	5463	21.42
1.95	98.05	14.1	14.6	6298	27.83	67083	140.93	23667	49.72	18575	72.56	4505	17.60
25.0	75.0	17.5	17.6	170658	775.72	58823	125.42	21240	45.29	55820	224.18	12972	52.10
26.0	74.0	17.1	17.0	195229	883.39	57200	123.81	21059	45.58	49565	197.47	13686	54.52

Table 16

## Concrete Test Series Concrete Batches: Elemental Signature Intensities

Mix Proportions (%)				Aggregate Type*	Test No.	Sample Weights (lbs)		E L E M E N T A L   S I G N A T U R E   I N T E N S I T I E S									
Water	Cement	Fine Agg	Coarse Agg			TNC-ACC	FNC	N (TNC)		SI (FNC)		C (FNC)		AI-SI (ACC)		Ca (ACC)	
								Net	Net PHA T	Net	Net PHA T	Net	Net PHA T	Net	Net PHA T	Net	Net PHA T
9.83	21.50	26.7	41.8	S-S	1a	22.2	22.1	53543	240.1	185412	403.9	12580	27.4	266787	1028.9	7133	29.1
					1b	22.4	22.3	53724	242.0	193904	423.4	13740	30.0	273771	1126.6	7908	32.5
9.55	18.20	30.0	41.9	S-S	2a	22.0	22.0	51496	230.9	202763	440.8	13150	28.6	251603	1022.8	5775	23.5
					2b	22.3	22.2	48741	218.6	202516	441.2	13259	28.9	253148	1029.1	6313	25.7
10.19	15.90	31.7	42.3	S-S	3a	21.6	21.5	54017	242.2	203164	441.7	13176	28.6	240409	973.3	5574	22.6
					3b	21.6	21.8	50997	228.7	201130	438.2	13326	29.0	256128	1041.2	5677	23.1
9.50	14.00	33.8	42.7	S-S	4a	21.5	21.4	49201	221.6	207824	451.8	12852	27.9	217352	876.4	4649	18.7
					4b	21.8	21.7	47786	215.3	205958	448.7	14483	31.6	223695	909.3	5231	21.3
9.91	12.45	35.0	42.6	S-S	5a	21.6	21.2	57365	257.2	208350	452.9	12594	27.4	252245	1021.2	5522	22.4
					5b	21.9	21.6	54796	245.7	211394	459.6	13703	29.8	244447	989.7	4240	19.6
9.63	21.5	26.4	42.4	S-C	6a	22.5	22.6	46026	207.3	123865	268.7	17185	37.3	140499	566.5	10320	41.6
					6b	22.6	22.2	46045	206.5	118471	257.0	17560	38.1	145884	590.6	10804	43.7
9.52	18.2	29.6	42.7	S-C	7a	22.6	22.2	48803	218.8	130963	282.9	18039	39.0	143224	572.9	10653	42.6
					7b	22.4	22.6	47994	215.2	130638	282.8	17037	36.9	152467	614.8	10645	43.7
9.91	15.9	31.4	42.9	S-C	8a	22.2	22.2	51948	234.0	133378	290.6	16374	35.7	162436	657.6	12366	50.1
					8b	22.4	22.3	48191	215.1	129819	281.0	17574	38.0	155022	627.6	10007	40.5
9.41	14.0	33.7	42.9	S-C	9a	22.1	22.2	49292	218.1	141347	302.7	17043	36.5	150262	598.7	9744	38.8
					9b	22.3	22.2	55184	245.3	141047	302.0	17446	37.4	144608	580.8	9367	37.6
9.67	12.4	34.8	43.1	S-C	10a	22.4	22.5	44748	198.9	123588	265.8	6704	14.4	135903	543.6	9055	36.2
					10b	22.2	22.3	43027	191.2	144869	312.2	16606	35.8	137000	550.2	9706	39.0

\* Note: The first letter represents fine aggregate; the second letter represents coarse aggregate; S = siliceous, C = calcareous

Table 16 (Cont'd)

Mix Proportions (%)				Aggregate Type	Test Sample No.	Test Sample Weights (lbs)		ELEMENTAL SIGNATURE INTENSITIES											
Water	Cement	Fine Agg	Coarse Agg			TNC-ACC	FNC	H (TNC)		S1 (FNC)		C (FNC)		A1-S1 (ACC)		Ca (ACC)			
							Net	Net PHA T	Net	Net PHA T	Net	Net PHA T	Net	Net PHA T	Net	Net PHA T			
9.03	21.5	27.0	42.4	C-C	11a	22.3	47382	211.5	80803	174.1	20049	43.2	140602	564.7	12662	50.9			
					11b	22.5	46914	209.4	78469	169.1	20347	43.9	135657	544.8	12353	49.6			
8.99	18.2	30.0	42.8	C-C	12a	22.5	51588	231.3	74206	160.3	18065	39.0	139485	553.5	12319	48.9			
					12b	22.9	46024	205.5	72851	156.7	18713	40.2	141135	562.3	12761	50.8			
9.01	15.9	32.2	43.0	C-C	13a	22.6	51058	227.9	75365	162.4	18448	39.8	146889	587.6	12775	51.1			
					13b	22.9	47515	212.1	79355	171.0	19074	41.1	139503	560.3	11945	48.0			
9.76	14.0	33.2	43.1	C-C	14a	22.8	49691	220.8	78557	168.2	18889	40.4	160238	643.5	13350	53.6			
					14b	22.0	58791	262.5	73206	157.1	19495	41.8	151163	604.7	11403	45.6			
9.08	12.4	35.4	43.1	C-C	15a	22.2	48560	216.8	76439	164.4	18248	39.2	142416	569.7	11492	46.0			
					15b	21.9	46605	208.1	73774	158.7	20332	43.7	150509	602.0	11787	47.1			
9.46	21.5	26.6	42.3	C-C	16a	22.2	53935	241.9	144036	311.1	14561	31.4	228840	919.0	7980	32.0			
					16b	22.5	52129	233.8	150170	324.3	14644	31.6	231620	934.0	8669	35.0			
9.64	18.2	29.7	42.5	C-C	17a	22.5	53489	238.8	152535	330.2	15100	32.7	210317	851.5	7468	30.2			
					17b	22.1	49625	221.5	149113	322.8	15736	34.1	221020	894.8	7850	31.8			
9.49	15.9	31.8	42.9	C-C	18a	22.2	54980	246.5	143214	309.3	14849	32.1	232643	938.1	8541	34.4			
					18b	22.1	51857	232.5	142767	310.4	15417	33.5	255058	1036.8	8490	34.5			
10.22	14.0	32.9	42.9	C-C	19a	21.8	55744	248.9	148972	322.5	16298	35.5	214960	866.8	7816	31.5			
					19b	22.4	53398	239.5	148766	322.0	16592	35.9	232783	946.3	7769	31.6			
9.76	12.4	35.0	42.7	C-C	20a	22.2	53686	240.7	148673	321.8	17267	37.4	209759	849.2	6912	28.0			
					20b	22.2	51125	229.3	151292	327.5	15591	34.0	223587	904.4	7788	51.5			

The mixing procedure for the concrete tests consisted of weighing out batch proportions so that each batch contained approximately 2 1/2 cu ft (.05 m<sup>3</sup>), and then obtaining representative samples of the fine and coarse aggregates for moisture determination (ASTM C 566). The batches were mixed in a 3 1/2 cu ft (.07 m<sup>3</sup>) drum type mixer for 4 to 5 min.

#### *Test Procedure*

With one exception, the procedure used for the neutron/gamma tests on constituents was the same as that described in the mortar test section, i.e., filling the two sample containers, smoothing off the tops evenly, and determining the weight of each sample before testing. The exception was the saturated-immersed coarse aggregate samples, in which each container was filled level-full with saturated aggregates, and water added to fill the container to the top. The weights of the samples were then determined before irradiation of the samples. Immediately after the neutron/gamma test, the immersed samples were oven-dried until all water was driven off. The samples were then weighed again.

During the irradiation-counting cycle, the pulse height analysis (PHA) time\* for each neutron/gamma test was recorded. The PHA time was recorded at the initiation of irradiation, the end of the TNC cycle (5 min), the beginning of the ACC cycle (6 min), and at the end of the FNC cycle (10 min) and the ACC cycle (11 min).

After the irradiation-counting cycle for the constituent neutron/gamma test, the TNC spectra were analyzed for the 2.22 MeV H peaks, the FNC spectra for the 4.43 MeV C and the 1.78 MeV Si peaks, and the ACC spectra for the 3.09 MeV Ca and the 1.78 MeV Al peaks. The peak widths used to compute net peak intensities were the same as those used in the mortar test series, i.e., four channel for the 2.22 MeV H peak, six-channel for the 1.78 MeV Si peak, and eight-channel for the remainder. In addition, maximum net peak intensities and net peak intensities starting one channel on either side of the maximum were computed.

A modified Kelly-Vail cement content test was run on each aggregate type used in the concrete test series. The procedure consisted of running Kelly-Vail cement content tests on 325-g river and limestone

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\* The PHA time is the relative percentage of incoming signal that is being processed by the analyzer. The analyzer requires 1.5  $\mu$  seconds to process each incoming signal. When incoming signal rates are slower than 2/3 signals per  $\mu$  seconds, the PHA time and true time are equal. But when the incoming rate is faster than 2/3 signals per  $\mu$  seconds, only a certain percentage of the signal is processed. This relative percentage is the PHA time divided by the true time.



sand samples and 430-g 3/4 in. (1.90-cm) gravel and 3/4-in. (1.90-cm) limestone samples. The cement equivalency of each aggregate type was determined.

The test procedure for the concrete batches consisted of two complete neutron/gamma and two complete Kelly-Vail tests per batch. The air content (ASTM C 231), unit weight (ASTM C 138), and slump (ASTM C 143) of each batch were determined, and four 6- by 12-in. (15.2- x 30.4-cm) cylinders were cast. The cylinders were moist cured (ASTM C-511); two were capped and broken after 7 days and two after 28 days (ASTM C 39). Starting with a homogeneous 2 1/2 cu ft (.05 m<sup>3</sup>) of concrete, the test sequence was to fill the two 8 by 5 in. (20.3 x 12.7 cm) neutron/gamma sample containers, determine their weights, and initiate the first neutron/gamma test. Concurrently, a slump test was run, air content was determined by pressure meter, two 5- to 6-kg samples of concrete were placed in 5-qt (5.6-l) polyethylene tubs and covered (Kelly-Vail subsamples), and the four 6- by 12-in. (15.2- x 30.4-cm) cylinders were cast. After the first neutron/gamma test was completed, the two 8- by 5-in. (20.4- x 12.7-cm) sample containers were emptied, cleaned, refilled, weighed, and the second neutron/gamma test was initiated. Following the casting of the 6- by 12-in. (15.2- x 30.4-cm) cylinders, the Kelly-Vail tests were begun. Each Kelly-Vail subsample was handmixed to insure homogeneity before the test. For details of the Kelly-Vail and neutron/gamma operating procedures, see Chapter 3.

The concrete test series collected the same spectral analysis data and used the same procedure to determine net peak intensities as was used for the mortar and concrete constituent tests. The collected data were the net peaks for the 2.22 MeV  $H(n, \gamma)H$ , 1.78 MeV  $^{28}Si(n, n')^{28}Si$ , 4.43 MeV  $^{12}C(n, n')^{12}C$ , 3.09 MeV  $^{48}Ca(n, \gamma)^{49}Ca$ , and the 1.78 MeV from both  $^{27}Al(n, \gamma)^{28}Al$  and  $^{28}Si(n, p)^{28}Al$  reactions. As described earlier, maximum net peaks and the net peak for one channel on each side were computed to insure that the maximum net peak had been obtained. Only maximum net peak (signature) intensities were used in the data analysis. The PHA times for each test were also recorded.

#### *Test Results*

Tables 13, 14, and 15 list the signature intensities and signature intensities divided by the PHA (rate intensities) times for the neutron/gamma tests on the concrete material constituents. Table 16 lists the signature intensities and rate intensities for the neutron/gamma tests on the four concrete test series. Figures 12 through 23 are typical TNC, ACC, and FNC spectra for the four concrete test series. Table 17 lists the slump, unit weight, air content, compressive strengths, and Kelly-Vail test results for the four concrete test series.

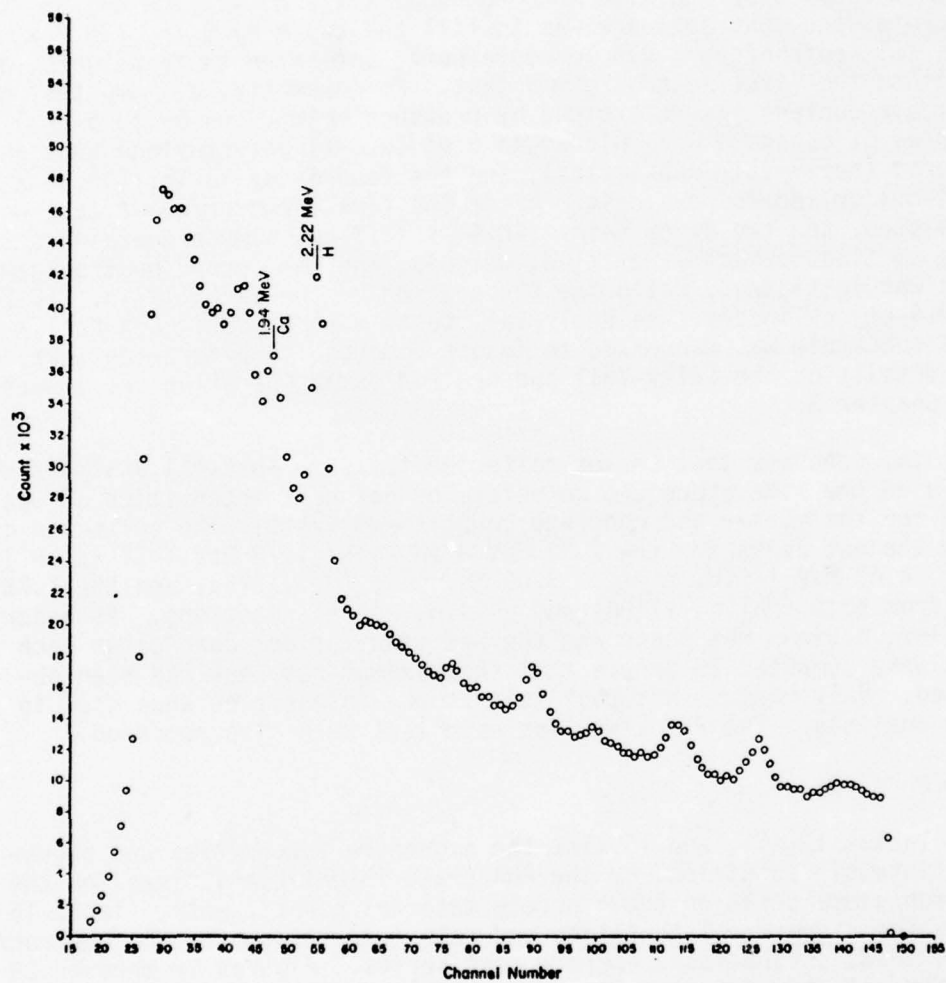


Figure 12. TNC spectrum, mix no. 3, river sand - 3/4 in. (1.90 cm) gravel concrete.

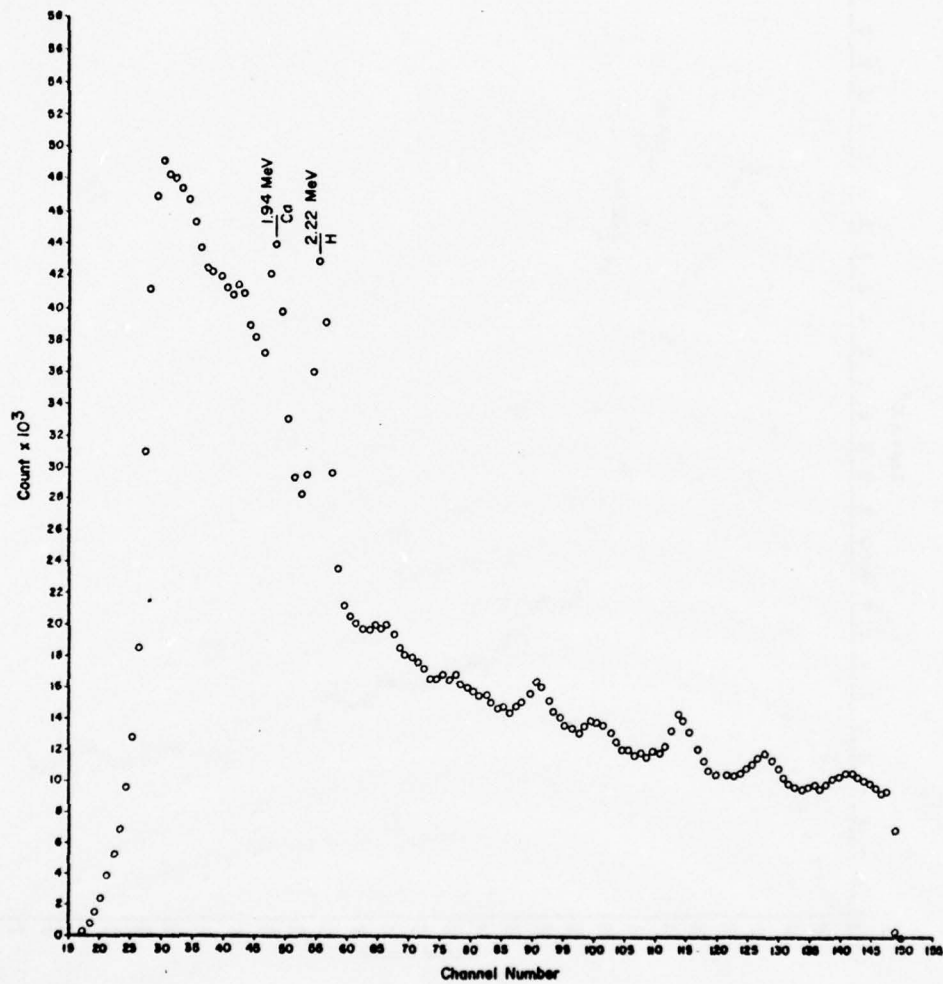


Figure 13. TNC spectrum, mix no. 8, river sand - 3/4 in. (1.90 cm) limestone concrete.

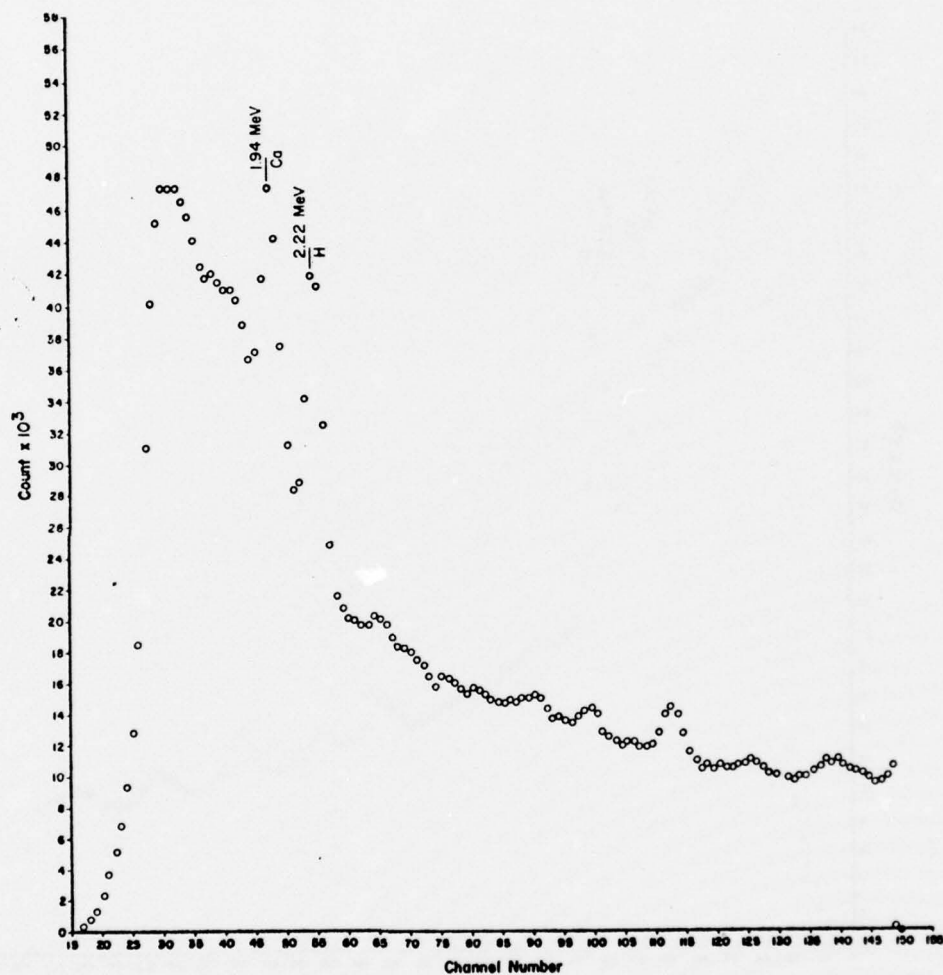


Figure 14. TNC spectrum, mix no. 13, limestone sand - 3/4 in. (1.90 cm) limestone concrete.



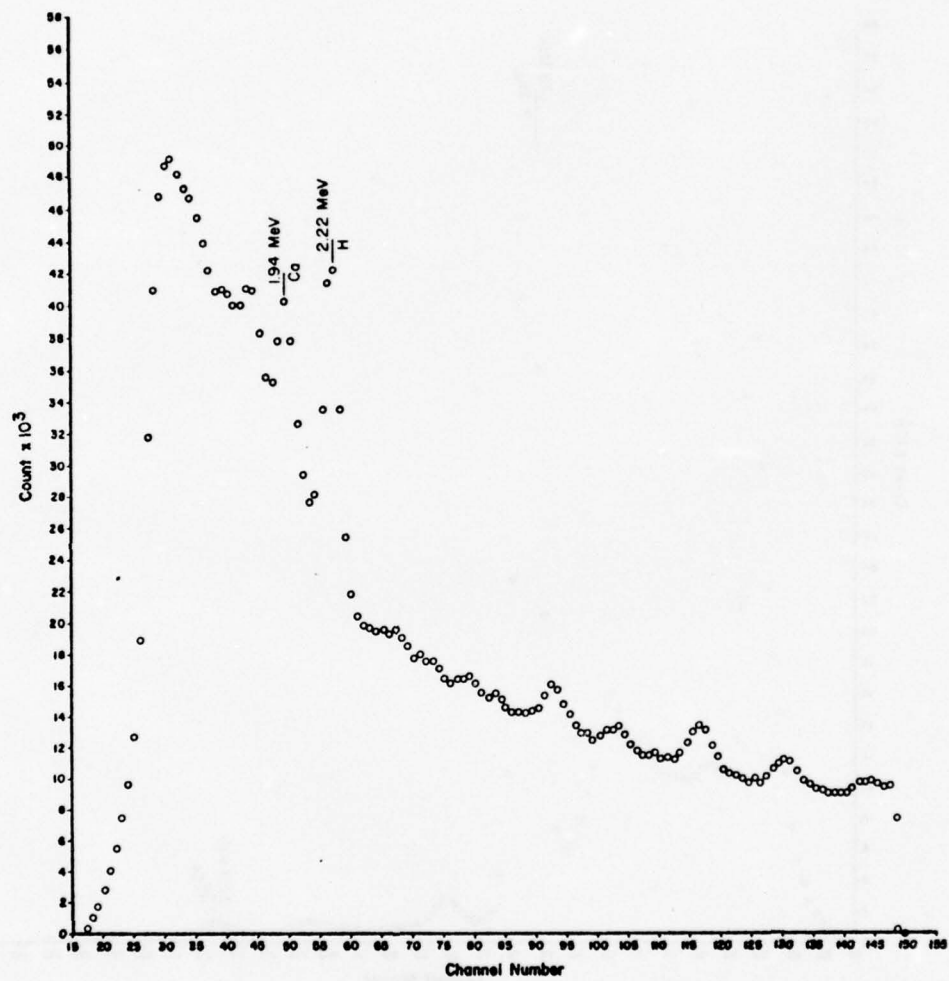


Figure 15. TNC spectrum, mix no. 18, limestone sand - 3/4 in. (1.90 cm) gravel concrete.

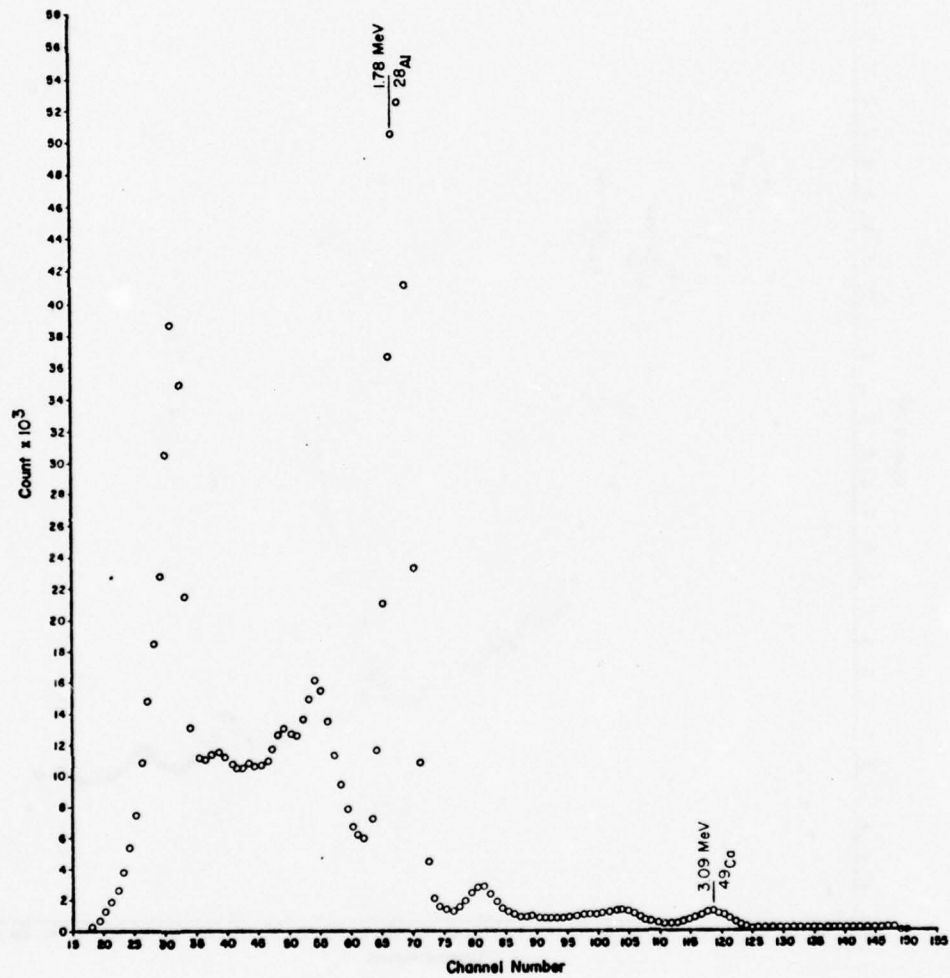


Figure 16. ACC spectrum, mix no. 3, river sand - 3/4 in. (1.90 cm) gravel concrete.

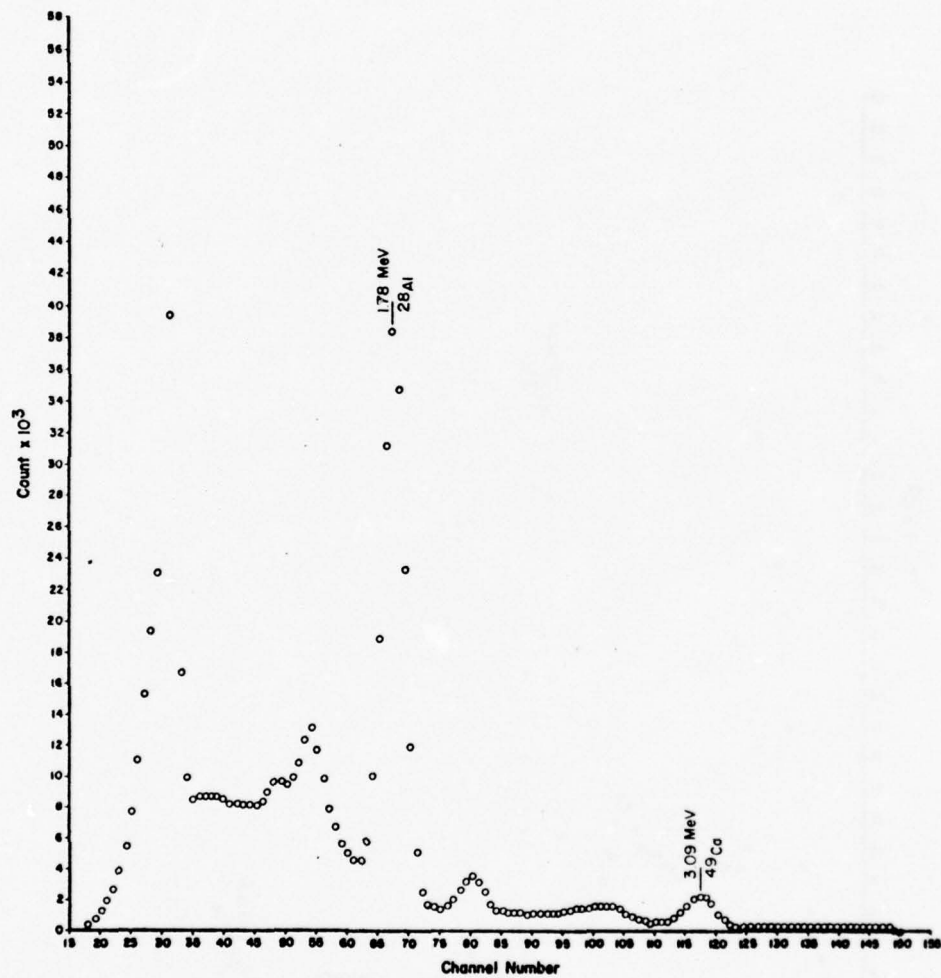


Figure 17. ACC spectrum, mix no. 8, river sand - 3/4 in. (1.90 cm) limestone concrete.

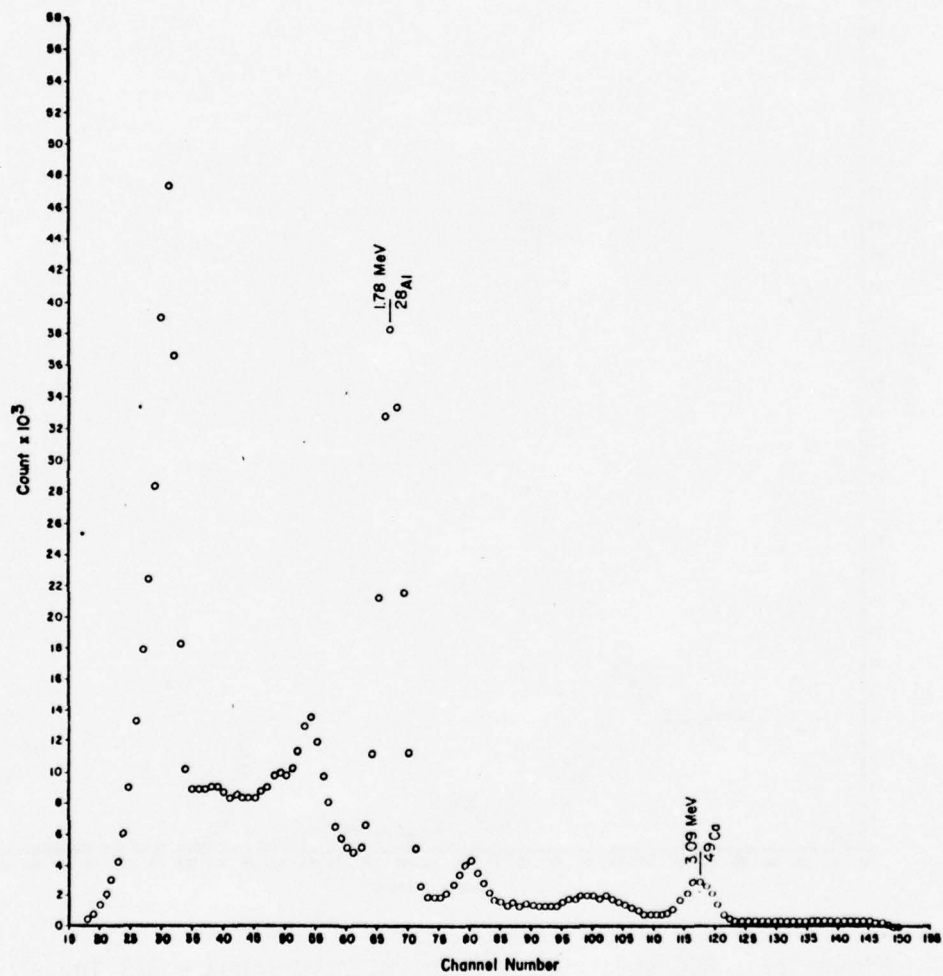


Figure 18. ACC spectrum, mix no. 13, limestone sand - 3/4 in. (1.90 cm) limestone concrete.



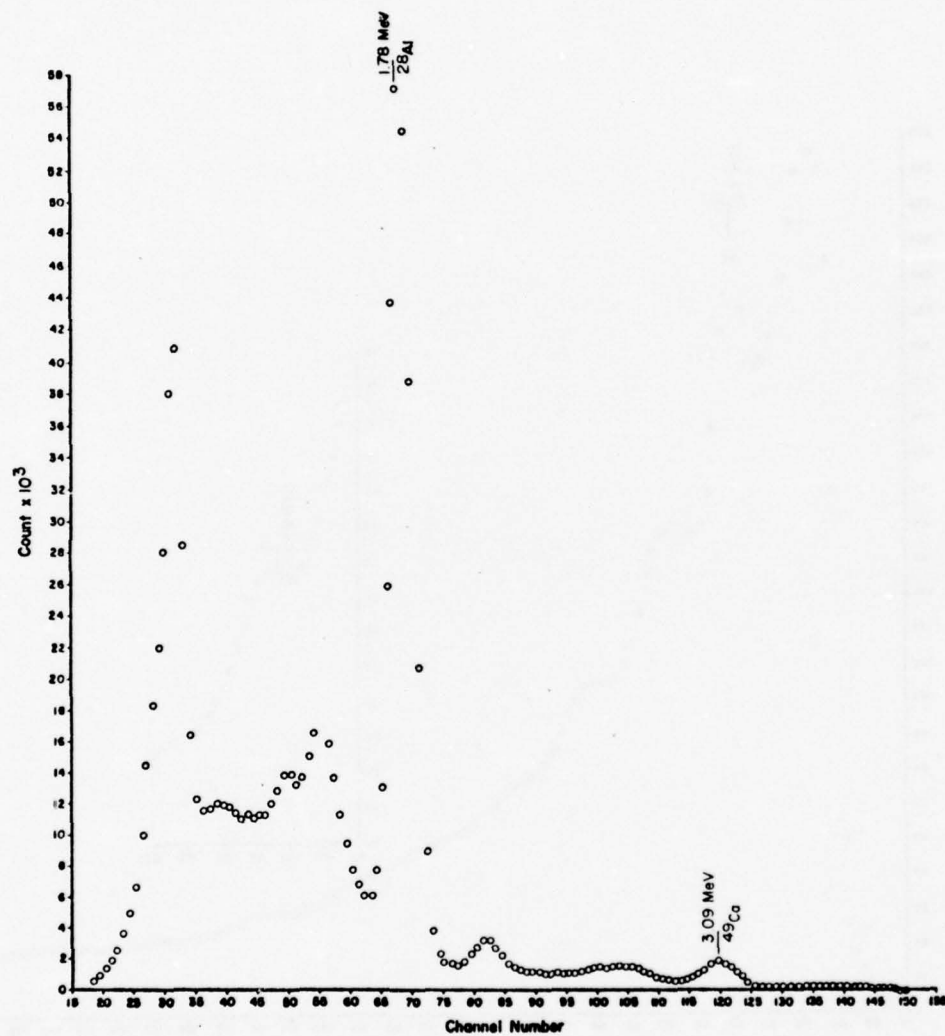


Figure 19. ACC spectrum, mix no. 18, limestone sand - 3/4 in. (1.90 cm) gravel concrete.

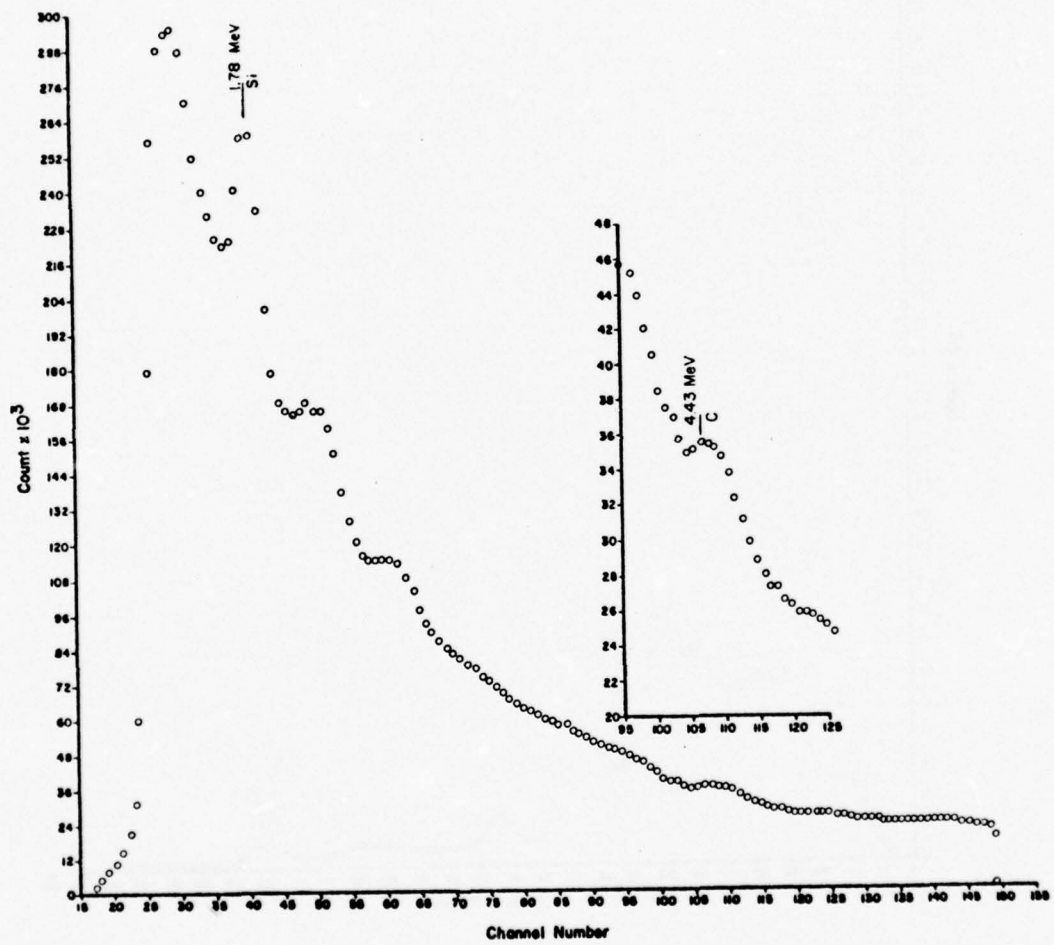


Figure 20. FNC spectrum, mix no. 3, river sand - 3/4 in. (1.90 cm) gravel concrete.

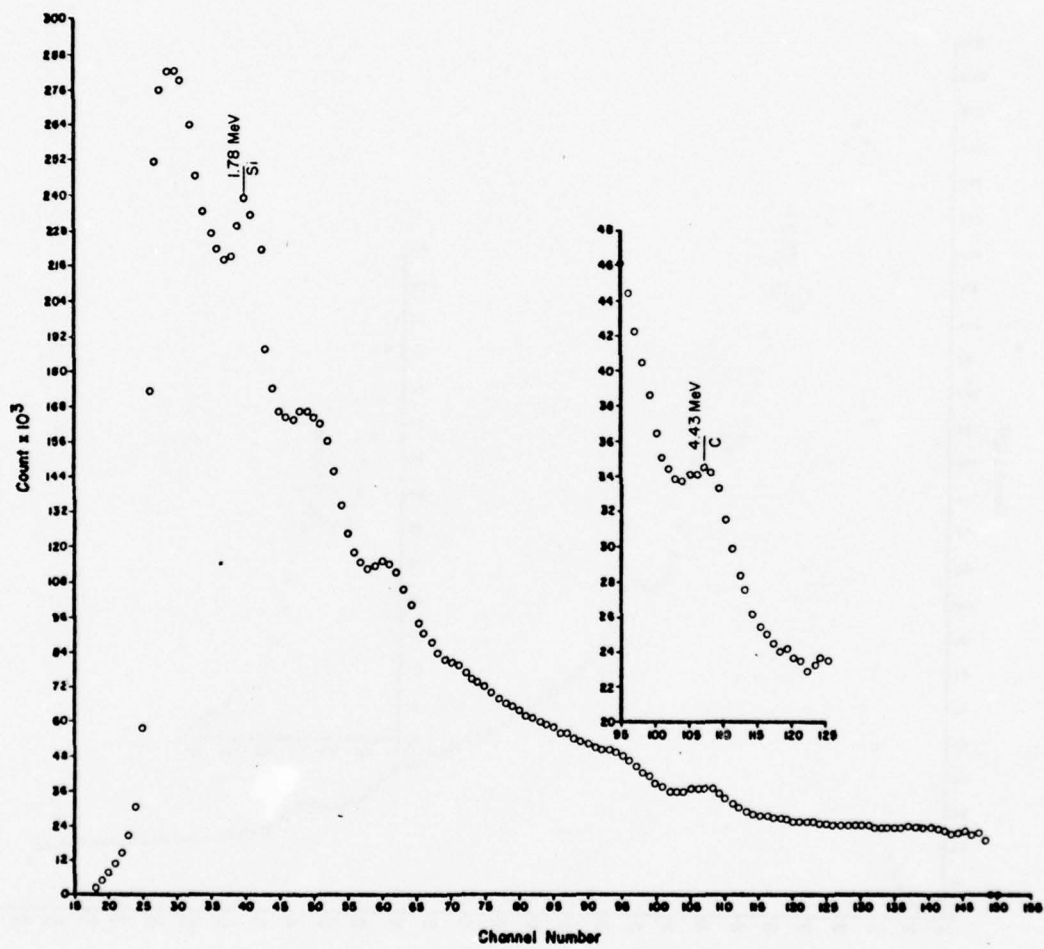


Figure 21. FNC spectrum, mix no. 8, river sand - 3/4 in. (1.90 cm) limestone concrete.

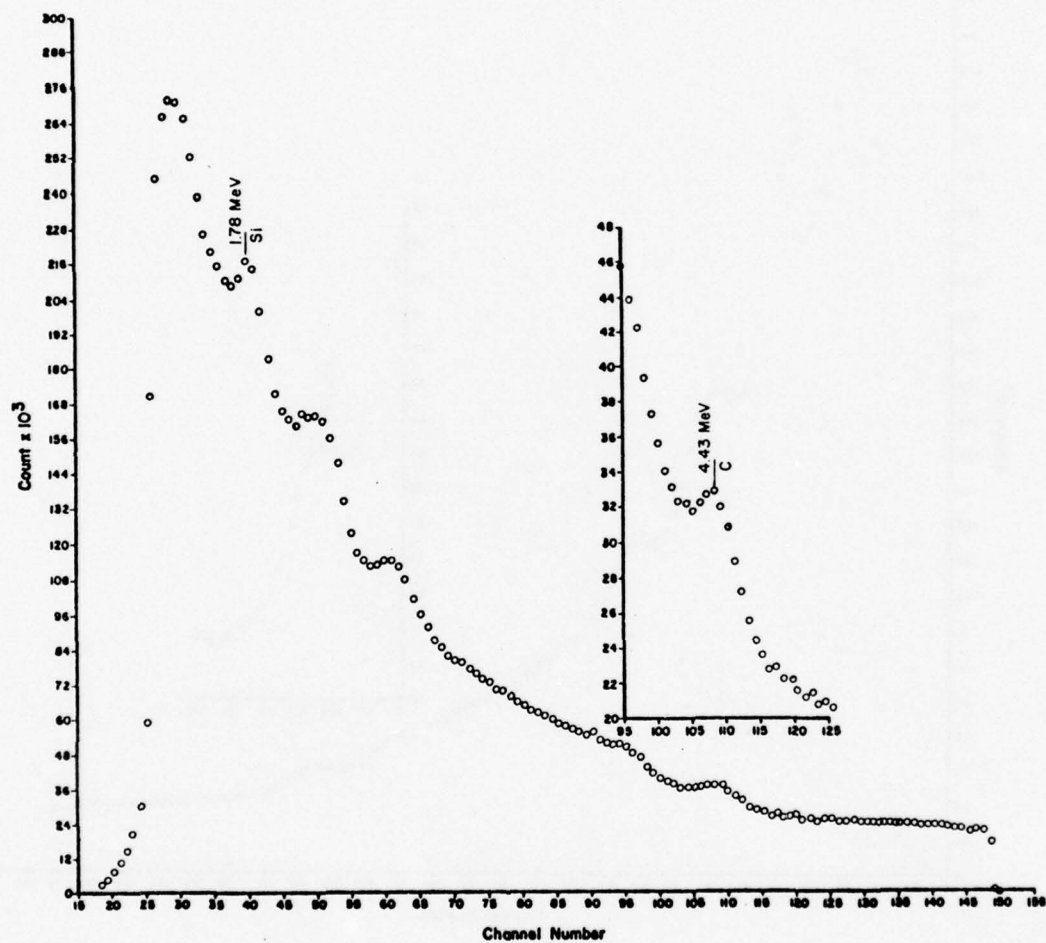


Figure 22. FNC spectrum, mix no. 13, limestone sand - 3/4 in. (1.90 cm) limestone concrete.



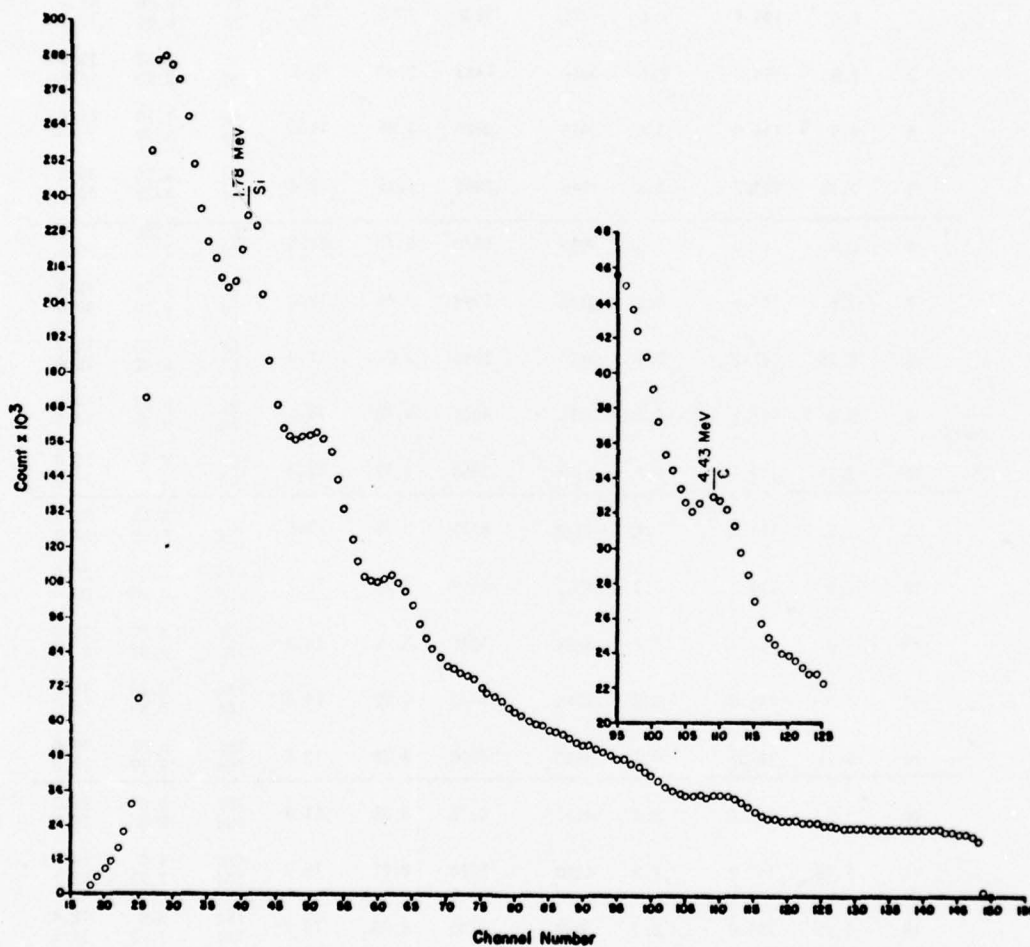


Figure 23. FNC spectrum, mix no. 18, limestone sand - 3/4 in. (1.90 cm) gravel concrete.

Table 17

Concrete Test Series--Data on Concrete Tests  
Kelly-Vail, Strength, Unit Weight, Slump

Batch No.	Slump Unit		Air %	Comp. Strength		Actual		Test No.	Kelly/Vail	
	(in)	Wt. (lbs/ft <sup>3</sup> )		Avg (psi) 7 day	28 day	Water (%)	Cement (%)		Water (%)	Cement (%)
1	4.5	146.0	22	4530	5782	8.28	21.5	1c 1d	8.28 8.00	20.1 19.7
2	6.0	146.4	2.6	4275	5632	7.95	18.2	2c 2d	8.28 8.00	17.4 17.7
3	5.5	144.8	2.6	3042	4483	8.58	15.9	3c 3d	8.55 9.10	15.8 15.6
4	5.5	144.0	3.4	2445	3520	7.85	14.0	4c 4d	7.75 8.28	13.6 13.8
5	7.75	143.2	2.8	1845	2865	8.25	12.4	5c 5d	9.10 8.28	13.2 12.4
6	8.0	148.0	1.5	4845	5670	8.71	21.5	6c 6d	8.82 8.28	21.3 20.6
7	7.5	147.6	0.9	4120	5394	8.64	18.2	7c 7d	9.28 9.10	18.4 18.0
8	9.25	147.2	0.9	3450	5090	9.01	15.9	8c 8d	8.28 8.82	14.7 15.8
9	9.0	147.2	0.8	3015	4065	8.48	14.0	9c 9d	8.82 9.35	14.3 14.2
10	8.75	145.6	0.8	2378	3350	8.72	12.4	10c 10d	9.4 9.4	13.0 12.4
11	3.5	149.2	1.4	4730	6335	8.33	21.5	11c 11d	8.55 8.55	23.2 24.2
12	6.5	149.2	1.3	4705	6550	8.27	18.2	12c 12d	8.00 8.28	21.5 20.8
13	5.0	148.4	1.1	3655	5245	8.28	15.9	13c 13d	8.82 8.82	20.2 21.8
14	7.5	149.6	0.6	3045	4445	9.02	14.0	14c 14d	8.28 8.55	16.8 16.5
15	4.25	147.2	1.2	2690	4100	8.32	12.4	15c 15d	8.82 9.10	19.6 17.8
16	3.5	148.4	2.2	4527	6070	8.05	21.5	16c 16d	8.0 8.0	26.0 23.8
17	5.75	145.0	2.5	4210	5770	8.21	18.2	17c 17d	8.0 7.72	19.0 22.1
18	4.75	145.6	2.3	3059	4730	8.03	15.9	18c 18d	8.0 7.72	18.4 17.5
19	6.75	145.6	2.1	2740	4130	8.76	14.0	19c 19d	8.28 8.28	16.5 17.2
20	6.25	145.2	2.3	2250	3635	8.30	12.4	20c 20d	8.82 8.28	16.1 16.2

## 5 ANALYSIS AND DISCUSSION OF TEST RESULTS

### Analysis of Signature-Constituent Responses

The objective of the signature-constituent analysis is to determine: (1) the linearity between signature intensities and the associated elemental intensities of the test samples; (2) the sensitivity of the signatures to other elements (matrix effects) in the test samples; (3) the relationship between the signatures for individual constituent samples (cement, fine aggregates, and coarse aggregates) and their associated signatures when combined and in the presence of water; and (4) the theoretical and operational accuracy of each signature. Appendix A provides details of the signature-constituent analysis; the following summarizes the analysis results.

The H signature is linearly related to the sample's water content; however, the study could not determine conclusively whether the H signature/water content relationship was related to the relative percentage of H (water) present in the sample or to the quantity of H (water) present. The sensitivity of the H signature to other elements in the test sample is minimal if a four-channel net peak is used. For all materials used in this study except the polyester samples, the H signature is almost exclusively related to the sample's water content. The theoretical error in the H signature for concrete type materials was approximately 1 percent, and the operational error based on repetitive tests on a given stable sample varied from less than 1 to more than 3 percent.

The data indicate that the Ca signature (and signature rate intensity) is linearly related to the sample's Ca content. The sensitivity of the Ca signature to elements common to concrete other than H (water) appears to be negligible. The signature's sensitivity to H is significant when anhydrous samples are compared to samples containing water, but when compared to the range of water contents common to mortars and concrete, the signature's sensitivity to H is negligible. There does not appear to be any single or simple relationship between the Ca signatures for individual dry constituents (cement, fine aggregates, and coarse aggregates) and their associated signatures when combined with water. The theoretical error in the Ca signature for concrete type materials varied from 1.0 to 2.3 percent, and the operational error based on repetitive tests on a given stable sample varied from 1.5 to 3.0 percent.

The Al-Si signature (signature rate intensity) is linearly related to the test sample's Al plus Si content; however, since the signature sensitivity to the two different reactions is different and varies relative to the sample's H (water) content, the resulting

signature is complex and cannot be related simply to either Al or Si. The sensitivity of the Al-Si signature to elements other than H (water) appears to be negligible. The signature sensitivity to H is only significant when Al is present and when anhydrous samples are compared to samples containing water. For the variations in water content common to concretes and mortars, the signature's sensitivity to H is negligible. Due to the complex nature of the Al-Si signature and the H influence, there is no single or simple relationship between the Al-Si signatures for individual dry constituents (cement, fine aggregate, and coarse aggregate) and their associated signatures when combined with water. The theoretical error in the Al-Si signature for concrete type materials varied from 0.22 to 0.33 percent, and the operational error based on repetitive tests on a stable sample varied from 1 to 2 percent. Two dominant factors can be observed from the C-signature analysis: (1) a large percentage of the C signature is from the surrounding media and is not associated with the sample being tested; and (2) if the background C is removed from the net signature, the theoretical accuracy of the C signature for concrete type materials ranges from 7.6 to 40 percent. This level of accuracy is unacceptably low and makes further analysis of the C signature almost meaningless.

The data indicate that the Si signature is linearly related to the Si content of the sample. The Si signature is not sensitive to other elements common to concrete. On a percentage constituent basis, there appears to be a direct correlation between the individual dry constituent and the constituents' associated signatures when combined with water. The theoretical error in the Si signature varied from 0.77 to 2.29 percent, and the operational error based on repetitive tests of a given sample varied from 1.34 to 3.2 percent.

#### Neutron/Gamma Estimates of Water and Cement Contents

Eq 1 (Chapter 3) is used to compute the water and cement contents of the mortar and concrete mixes. Using the fact that the H signature is unique to water content, Eq 1 simplifies to a linear relationship between H signature and water content (total water) and a three by three matrix for cement content (concrete). Any three of the four remaining signatures can be used in the cement matrix equation. The mortar test requires only a two by two matrix equation for cement content.

The multipliers used to compute the cement contents of the mortars were those obtained from the mortar regression analysis (see Appendix). Water contents for the mortars were obtained directly from the linear regressions on the mortar data relating the H signature to water content percent. Table 18 contains the results of the computed water and cement contents for the Ottawa sand mortars. Only two unique solutions to cement content can be computed, since Ca is unique to the cement and there is no C present in either the cement or Ottawa sand.



Table 18  
Computed Water and Cement Contents--Ottawa Sand Mortar Data

Actual Mix Proportions			Computed Mix Proportions					
Water %	Cement %	Sand %	Ca		Al-Si/Si		H*	
			Cement	Recovery	Cement	Recovery	Water	Recovery
7.9	23.0	69.0	21.2	91.9	20.8	90.4	7.81	98.9
9.1	22.7	68.1	20.8	91.6	22.1	97.4	9.00	98.9
10.4	22.4	67.2	22.7	101.2	23.5	105.1	10.1	97.1
7.9	18.4	73.7	18.5	100.6	18.7	101.7	8.05	101.9
9.1	18.2	72.7	18.6	102.5	20.00	109.8	8.95	98.4
10.4	17.9	71.7	18.2	101.5	18.1	101.4	10.7	102.9
7.9	13.2	79.0	12.4	93.9	11.3	85.5	8.14	103.0
9.1	13.0	77.9	15.2	116.9	15.2	116.8	9.03	99.3

$\bar{x} = 100.0$        $\bar{x} = 101$        $\bar{x} = 100$   
 $S_x = 8.2$        $S_x = 10.1$        $S_x = 2.23$

\* % H<sub>2</sub>O = 3.47 + 1.36 4 x 10<sup>-4</sup> H count

The one solution relates the Ca signature directly to cement content. The other is the matrix solution using the Al-Si and Si signatures. If recovery (predicted divided by actual) is used as the basis of comparison, cement recovery based on the Ca signature has a standard deviation of 8.2 percent. Cement recovery from the Al-Si/Si matrix solution has a standard deviation of 10.1 percent. The standard deviation for the water recovery relating the H signature to water content is 2.23 percent.

Table 19 contains the computed water and cement contents for the river sand mortars. Of the six possible signature combinations for computing cement content, it is noted that the Ca signature is the key to obtaining reasonably accurate results. The three CA solutions (Ca/Al-Si, Ca/C, and Ca/Si) have standard deviations for cement recovery varying from 11 to 11.8 percent. The three other combinations (Al-Si/C, Al-Si/Si, and C/Si) produced standard deviations varying from 17.7 to 40.6 percent. The standard deviation for water recovery is 2.5 percent.

Table 20 contains the computed water and cement contents for the limestone sand mortars. In this test, equipment malfunctions occurred and the Si signature was not obtained accurately, so only three unique solutions for cement content could be computed: Ca/Al-Si, Ca/C, and Al-Si/C. Their respective standard deviations for cement recovery were 23.6, 42.5, and 23.3 percent. The standard deviation for the water recovery was 2.1 percent.

The water and cement contents of the concretes were computed on both a percent and weight basis. Water contents were computed from the three regression equations illustrated in Figure 24 and the three in Figure 25. The equations in Figure 24 are in terms of water content percent, and the equations in Figure 25 are in terms of water content weight. The three regression equations of each figure are based on: (1) the combined constituent data; (2) the concrete data; and (3) the modified concrete data (an equal number of data points assumed at the origin).

Tables 21 and 22 list the computed water contents on a percent and weight basis, respectively. The tables also list the related recovery values and means and the associated standard deviations for recovery. The means and standard deviations are computed for each of the four concrete test series and for all four series combined. The overall mean recoveries based on the percent computations (Table 21) ranged from 90.6 percent for regression curve number 1 to 100.8 percent for regression curve number 2. The overall mean recoveries based on weight computations (Table 22) ranged from 69.6 percent for regression curve number 1 to 100.2 percent for regression curve number 2. A comparison of the two tables indicates that when the constituent-water regression curves are used to estimate the water contents of the concretes, the percent relationship is the most accurate.

Table 19  
Computed Water and Cement Contents--River Sand Mortars

Actual Mix Proportions				Computed Mix Proportions												H*
Water %	Cement %	Sand %		Ca/Al-Si	Ca/C	Ca/Si	Al-Si/C	Al-Si/Si	C/Si	Ca/Al-Si	Ca/C	Ca/Si	Al-Si/C	Al-Si/Si	C/Si	
				Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	Cement Recovery	
7.9	23.0	69.0		19.4	84.2	20.1	87.2	19.2	83.5	14.8	64.5	18.6	80.8	17.2	74.9	7.85 99.4
9.1	22.7	68.1		23.6	104.2	26.3	111.5	23.4	103.3	6.31	27.8	22.6	99.4	16.7	73.4	9.43 103.6
10.4	22.4	67.2		22.9	102.4	22.2	98.9	22.5	100.3	28.0	125.0	20.5	91.3	23.2	103.6	103.5 99.6
7.9	18.4	73.7		20.0	108.8	19.9	108.4	19.6	106.4	20.6	112.2	17.6	95.7	18.7	101.7	7.69 97.4
9.1	18.2	72.7		17.8	98.0	18.0	98.8	18.0	99.2	17.0	93.2	18.9	104.1	18.2	100.2	8.99 98.8
10.4	17.9	71.7		18.4	102.7	18.3	102.2	18.6	103.6	19.0	106.0	19.3	107.6	19.2	107.1	10.04 96.5
7.9	13.2	79.0		10.7	81.4	9.8	74.4	10.8	81.8	16.7	126.8	11.0	83.6	13.1	99.2	8.18 103.6
9.1	13.0	77.9		14.0	107.8	13.4	103.1	14.3	109.8	17.9	137.7	15.4	118.5	16.3	125.5	9.12 100.2
10.4	12.8	76.8		14.6	114.5	13.7	106.9	15.2	118.9	21.0	163.9	17.6	137.5	18.8	147.1	10.53 101.2
				$\bar{x} = 100.4$		$\bar{x} = 99$		$\bar{x} = 100.8$		$\bar{x} = 106.3$		$\bar{x} = 102.1$		$\bar{x} = 103.6$		$\bar{x} = 100$
				$S_x = 11.0$		$S_x = 11.6$		$S_x = 11.8$		$S_x = 40.6$		$S_x = 17.7$		$S_x = 22.8$		$S_x = 2.46$

\* H =  $.65 \times 2.02 \times 10^{-4}$  H (count)

Table 20  
Computed Water and Cement Contents--Limestone Sand Mortars

Actual Mix Proportions			Computed Mix Proportions									
Water %	Cement %	Sand %	Ca/Al-Si			Ca/C			Al-Si/C			H*
			Cement	Recovery	Cement	Cement	Recovery	Cement	Recovery	Cement	Recovery	
7.9	23.0	69.0	23.3	101.5	19.8	19.8	86.3	22.7	98.7	7.8	99.2	
9.1	22.7	68.1	19.1	84.3	47.2	47.2	207.7	24.2	106.5	9.2	100.7	
10.4	22.4	67.2	19.2	85.8	28.3	28.3	126.5	20.9	93.2	10.0	96.0	
7.9	18.4	73.7	18.1	98.6	27.4	27.4	149.0	19.8	107.6	7.72	97.8	
9.1	18.2	72.7	19.4	106.6	36.5	36.5	200.4	22.5	123.5	9.1	100.1	
10.4	17.9	71.7	19.0	106.3	29.5	29.5	165.0	20.9	116.9	10.5	100.7	
7.9	13.2	79.0	14.7	111.8	27.2	27.2	206.4	17.0	128.8	8.1	102.6	
9.1	13.0	77.9	20.0	153.9	26.6	26.6	204.7	21.2	163.0	9.3	102.4	
10.4	12.8	76.8	8.9	69.8	19.4	19.4	152.2	10.8	84.6	10.5	100.8	

$\bar{x} = 100.0$   
 $S_x = 2.11$

$\bar{x} = 113.6$   
 $S_x = 23.2$

$\bar{x} = 166.4$   
 $S_x = 42.5$

$\bar{x} = 102.1$   
 $S_x = 23.6$

\*  $H_2O = 1.47 + 1.81 \times 10^{-4} H$  (count)



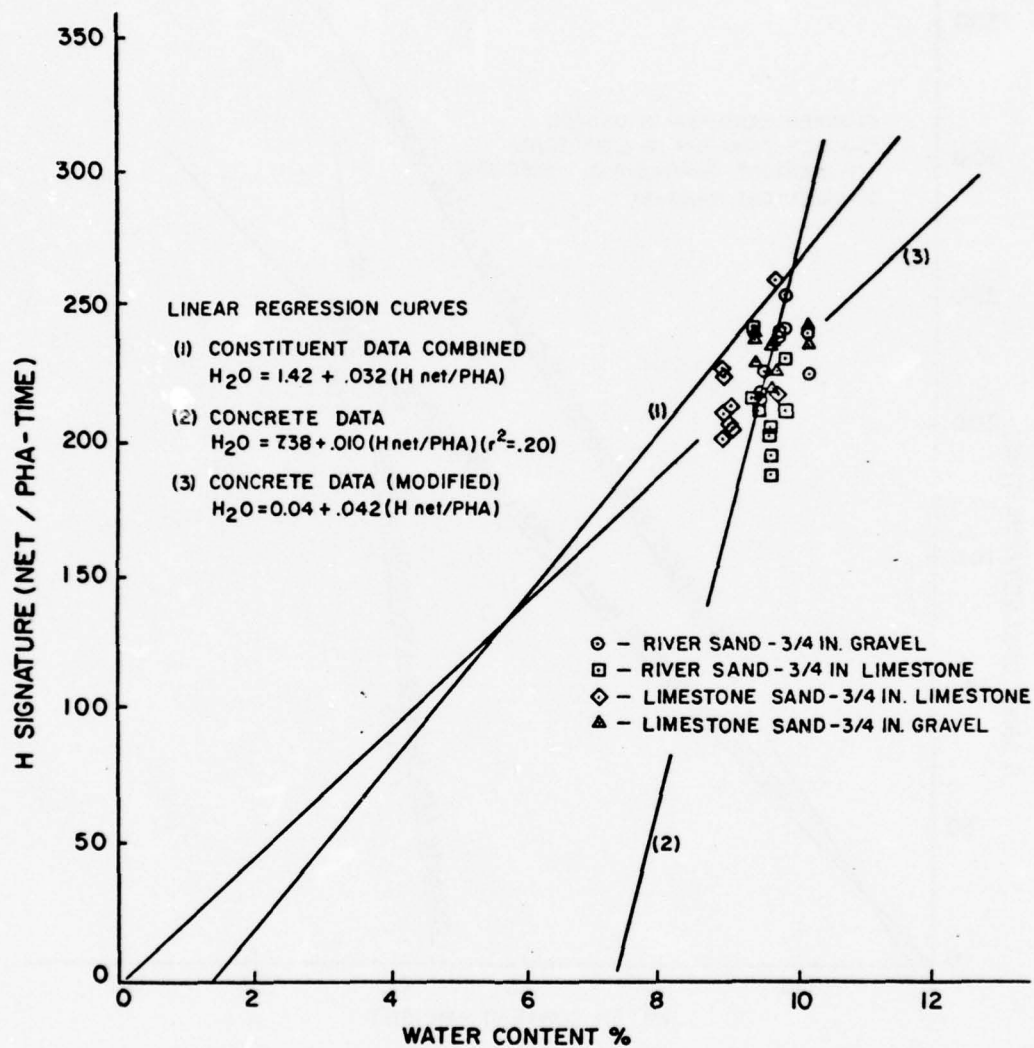


Figure 24. Water content vs. hydrogen signature content tests data (water content percent).

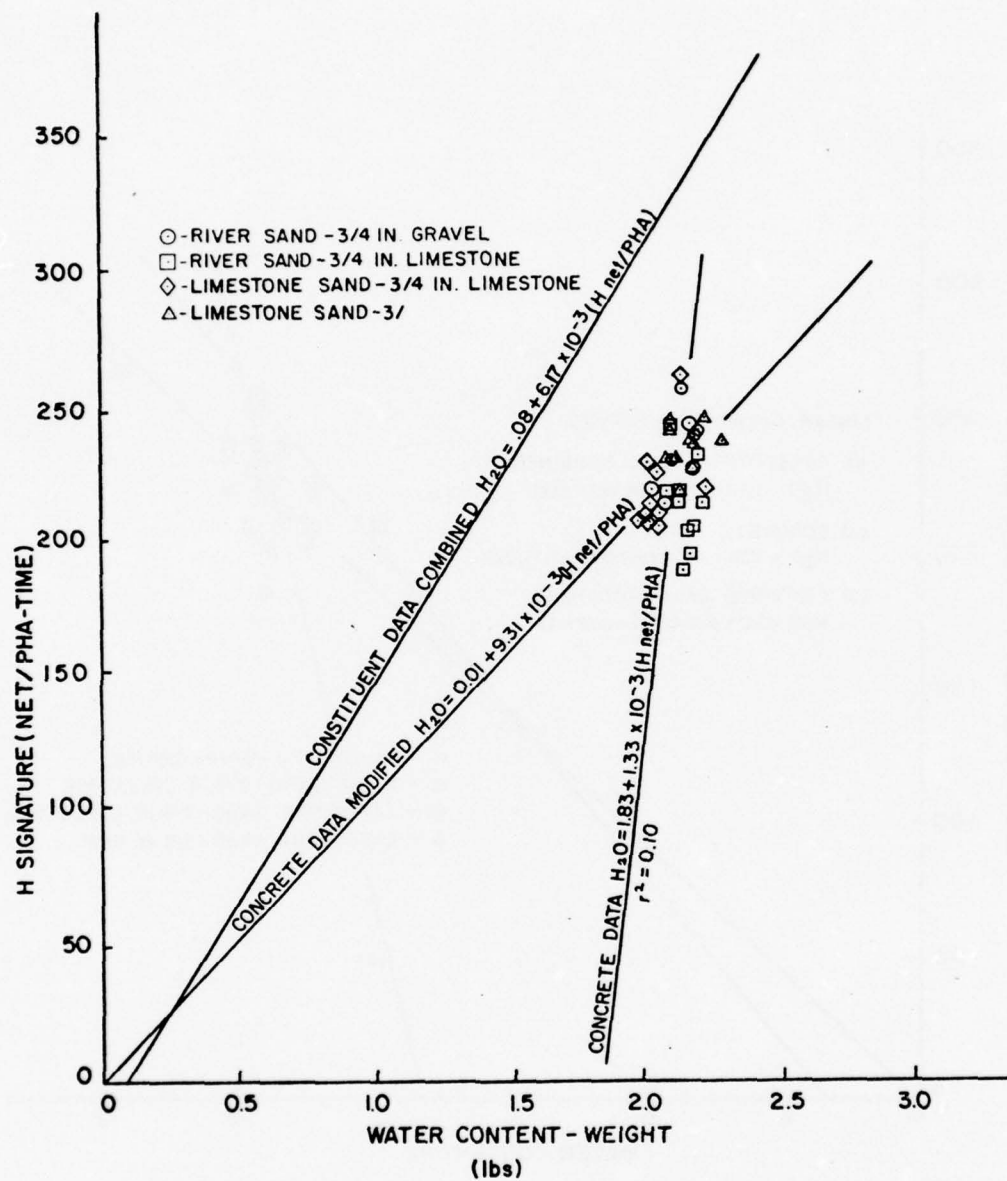


Figure 25. Water content vs. hydrogen signature concrete tests data (water content weight).

Table 21  
Water Contents--Concrete Data  
(Percent)

Test No.	Actual Water	Predicted Eq 1	Recovery Eq 1	Predicted Eq 2	Recovery Eq 2	Predicted Eq 3	Recovery Eq 3
1 1a.	9.83	9.10	92.6	9.78	99.5	10.12	103.0
1 1b.		9.16	93.2	9.80	99.7	10.20	103.8
2 2a.	9.55	8.81	92.2	9.69	101.4	9.74	102.0
2 2b.		8.42	88.2	9.57	100.2	9.22	96.6
3 3a.	10.19	9.16	89.9	9.80	96.2	10.21	100.2
3 3b.		8.74	85.8	9.67	94.9	9.64	94.6
4 4a.	9.50	8.51	89.6	9.60	101.0	9.35	98.4
4 4b.		8.31	87.4	9.53	100.3	9.08	95.6
5 5a.	9.91	9.65	97.4	9.95	100.4	10.84	109.4
5 5b.		9.28	93.7	9.84	99.3	10.36	104.5
		$\bar{x} = 91.0$		$\bar{x} = 99.3$		$\bar{x} = 100.8$	
		$S_x = 3.5$		$S_x = 2.1$		$S_x = 4.6$	
6 6a.	9.63	8.05	83.6	9.45	98.2	8.74	90.8
6 6b.		8.03	83.4	9.44	98.1	8.71	90.5
7 7a.	9.52	8.42	88.5	9.57	100.5	9.23	96.9
7 7b.		8.31	87.2	9.53	100.1	9.08	95.4
8 8a.	9.91	8.91	89.9	9.72	98.1	9.87	99.6
8 8b.		8.30	83.8	9.53	96.2	9.07	91.6
9 9a.	9.41	8.40	89.2	9.56	101.6	9.20	97.8
9 9b.		9.27	98.5	9.83	104.5	10.34	109.9
10 10a.	9.67	7.78	80.5	9.37	96.9	8.39	86.8
10 10b.		7.54	78.0	9.29	96.1	8.07	83.4
		$\bar{x} = 86.26$		$\bar{x} = 99.0$		$\bar{x} = 94.3$	
		$S_x = 5.76$		$S_x = 2.6$		$S_x = 7.5$	
11 11a.	9.03	8.12	89.9	9.47	104.9	8.84	97.8
11 11b.		8.19	90.7	9.49	105.1	8.92	98.8
12 12a.	8.99	8.82	98.1	9.69	107.8	9.75	108.5
12 12b.		8.00	88.9	9.44	104.9	8.61	96.4
13 13a.	9.01	8.71	96.7	9.66	107.2	9.61	106.7
13 13b.		8.21	91.1	9.50	105.4	8.95	99.3
14 14a.	9.76	8.48	86.9	9.59	98.2	9.31	95.4
14 14b.		9.82	100.6	10.01	102.5	11.06	113.4
15 15a.	9.08	8.36	92.0	9.55	105.2	9.14	100.7
15 15b.		8.08	89.0	9.46	104.2	8.78	96.7
		$\bar{x} = 92.4$		$\bar{x} = 104.5$		$\bar{x} = 101.4$	
		$S_x = 4.5$		$S_x = 4.0$		$S_x = 6.0$	
16 16a.	9.46	9.16	96.8	9.80	103.6	10.19	107.8
16 16b.		8.90	94.1	9.72	102.7	9.86	104.2
17 17a.	9.64	9.06	94.0	9.77	101.3	10.07	104.4
17 17b.		8.51	88.2	9.60	99.5	9.34	96.9
18 18a.	9.49	9.31	98.1	9.84	103.7	10.39	109.5
18 18b.		8.86	93.4	9.71	102.3	9.80	103.3
19 19a.	10.22	9.38	91.8	9.87	96.6	10.49	102.7
19 19b.		9.08	88.9	9.78	95.6	10.10	98.8
20 20a.	9.76	9.12	93.5	9.79	100.3	10.15	104.0
20 20b.		8.76	89.7	9.67	99.1	9.67	99.1
		$\bar{x} = 92.8$		$\bar{x} = 100.5$		$\bar{x} = 103.1$	
		$S_x = 3.2$		$S_x = 2.8$		$S_x = 3.94$	
		Total = 90.6		Total = 100.8		Total = 99.9	
		$S_x = 5.0$		$S_x = 3.3$		$S_x = 6.4$	
		C.V. = 5.6		C.V. = 3.3		C.V. = 6.5	

Table 22  
Water Contents--Concrete Data  
(Weight)

Test No.	Act.	Eq 1		Eq 2		Eq 3		
		Pred	Rec	Pred	Rec	Pred	Rec	
1	1a.	2.19	1.56	71.3	2.15	98.1	2.24	102.5
	1b.	2.21	1.57	71.2	2.15	97.4	2.26	102.4
2	2a.	2.10	1.50	71.6	2.14	101.8	2.16	102.8
	2b.	2.13	1.43	67.1	2.12	99.6	2.04	96.0
3	3a.	2.20	1.57	71.6	2.15	97.8	2.26	102.9
	3b.	2.19	1.49	68.1	2.13	97.5	2.14	97.6
4	4a.	2.04	1.45	70.9	2.12	104.2	2.07	101.6
	4b.	2.07	1.41	68.0	2.12	102.2	2.01	97.3
5	5a.	2.14	1.67	77.9	2.17	101.5	2.40	112.3
	5b.	2.17	1.60	73.5	2.16	99.4	2.30	105.8
			$\bar{x} = 71.1$		$\bar{x} = 100.0$		$\bar{x} = 102.1$	
			$S_x = 3.1$		$S_x = 2.4$		$S_x = 4.7$	
6	6a.	2.17	1.36	62.6	2.11	97.0	1.94	89.4
	6b.	2.18	1.35	62.1	2.11	96.5	1.93	88.6
7	7a.	2.15	1.52	70.9	2.12	98.7	2.05	95.2
	7b.	2.13	1.41	66.0	2.12	99.4	2.01	94.5
8	8a.	2.20	1.52	69.3	2.14	97.3	2.19	99.4
	8b.	2.22	1.41	63.4	2.12	95.3	2.01	90.6
9	9a.	2.10	1.59	75.9	2.16	102.7	2.29	109.2
	9b.	2.08	1.43	68.5	2.12	101.9	2.04	98.1
10	10a.	2.17	1.31	60.2	2.10	96.5	1.86	85.8
	10b.	2.15	1.26	58.6	2.08	96.9	1.70	83.2
			$\bar{x} = 65.8$		$\bar{x} = 98.2$		$\bar{x} = 93.4$	
			$S_x = 5.4$		$S_x = 2.4$		$S_x = 7.6$	
11	11a.	2.01	1.37	68.3	2.11	105.0	1.98	98.4
	11b.	2.03	1.38	68.2	2.11	103.9	1.96	96.5
12	12a.	2.02	1.51	74.6	2.14	105.8	2.16	107.1
	12b.	2.06	1.35	65.4	2.10	102.1	1.92	93.3
13	13a.	2.04	1.49	72.9	2.13	104.6	2.13	104.5
	13b.	2.06	1.39	67.4	2.11	102.5	1.98	96.3
14	14a.	2.23	1.44	64.7	2.12	95.2	2.07	92.6
	14b.	2.15	1.70	79.1	2.18	101.5	2.45	114.1
15	15a.	2.02	1.42	70.2	2.12	104.9	2.03	100.4
	15b.	1.99	1.36	68.5	2.11	105.9	1.95	97.8
			$\bar{x} = 69.9$		$\bar{x} = 103.1$		$\bar{x} = 100.1$	
			$S_x = 4.4$		$S_x = 3.2$		$S_x = 6.7$	
16	16a.	2.10	1.57	74.9	2.15	102.5	2.26	107.7
	16b.	2.13	1.52	71.5	2.14	100.5	2.19	102.6
17	17a.	2.17	1.55	71.6	2.15	99.0	2.23	102.9
	17b.	2.13	1.45	67.9	2.13	99.7	2.07	97.3
18	18a.	2.11	1.60	75.9	2.16	102.3	2.30	109.2
	18b.	2.10	1.52	72.1	2.14	101.9	2.17	103.5
19	19a.	2.23	1.62	72.5	2.16	96.9	2.33	104.3
	19b.	2.29	1.56	68.0	2.15	93.8	2.24	97.8
20	20a.	2.17	1.56	72.1	2.15	99.1	2.25	103.7
	20b.	2.17	1.49	68.9	2.14	98.4	2.14	98.8
			$\bar{x} = 71.5$		$\bar{x} = 99.4$		$\bar{x} = 102.8$	
			$S_x = 2.7$		$S_x = 2.7$		$S_x = 3.9$	
		Total = 69.7		Total = 100.2		Total = 99.6		
		$S_x = 4.5$		$S_x = 3.2$		$S_x = 6.8$		
		C.V. = 6.5		C.V. = 3.2		C.V. = 6.8		



Since curves 2 and 3 (Figures 24 and 25) were produced from the linear regressions on the concrete data, mean overall recoveries for both equations on each table were approximately 100 percent. The overall standard deviations for curve number 2 were 3.3 percent (Table 21) and 3.2 percent (Table 22). The overall standard deviations for curve number 3 were 6.4 percent (Table 21) and 6.8 percent (Table 22). The difference in accuracy for the two curves is expected, since curve 2 represents the best fit linear curve for the data, and curve 3 is a best fit curve although it must pass near the origin.

Even though curve 3 is a modified or forced relationship, it is probably more representative of the H signature water content relationship than curve 2. It is assumed that if the water contents of the concretes had varied as substantially as the water contents of the mortars and constituent water tests, curve 2 would have been similar to curves 1 and 3, and passed near the origin. The water content variations of the concrete tests were so small that the resulting variations in the H signatures were more closely related to H-signature accuracy than to any real variations in water content. Thus, in assessing the overall accuracy of the H signature-water content relationship, the 6.4 and 6.8 percent standard deviations from curve 3 are assumed to be the most realistic representation of accuracy.

The cement contents of the concretes were estimated from three sets of constituent-signature multipliers. The first two sets were obtained from the multiple linear regression analysis with upper and lower bounds on the concrete data. One was computed on a percent basis, and the other on a weight basis. The third set of multipliers was computed from the simple linear regression analysis, relating the signatures to variations in cement and fine aggregate content. These multipliers were computed only in terms of percent. For a detailed description of the three sets of constituent-signature multipliers, see the Appendix.

Since four signatures (C, Ca, Si, and Al-Si) were available when only three were required for a unique solution, four unique solutions for cement content were obtained from each set of multipliers for each concrete test.

Tables 23 through 26 are the cement, fine aggregates, and coarse aggregate contents computed for each of the four concrete test series using the percent multipliers from the multiple linear regression analysis. Similarly, Tables 27 through 30 are the cement, fine aggregate, and coarse aggregate contents computed by using the weight multipliers from the multiple linear regression analysis. Table 31 is the computed cement contents from the simple linear regression data for each of the four concrete test series. The computed constituent contents in Tables 21 through 30 are the average of the two neutron/gamma tests per mix. The computed cement contents in Table 31 are for each neutron/gamma test.

Table 23

Computed Constituent Contents - Percent  
River Sand - 3/4 Gravel - Concrete

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. %	Recovery	Pred. %	Recovery	Pred. %	Recovery
#1 Actual	--	21.5	--	26.7	--	41.8
C, Ca, Al-Si	24.70	114.8	42.25	158.2	38.04	91.0
C, Al-Si, Si	36.42	169.4	19.55	73.22	43.70	104.5
C, Ca, Si	24.75	115.1	24.52	91.8	42.46	101.6
Ca, Al-Si, Si	15.85	73.7	-0.46	-1.7	90.07	215.5
Average		118.2		80.4		128.2
#2 Actual	--	18.2	--	30.0	--	41.9
C, Ca, Al-Si	17.56	96.5	44.21	147.4	37.55	89.6
C, Al-Si, Si	25.55	140.4	28.74	95.8	41.41	98.8
C, Ca, Si	17.60	96.7	32.12	107.1	40.56	96.8
Ca, Al-Si, Si	11.52	63.3	15.10	50.3	73.0	174.2
Average		99.2		100.15		114.8
#3 Actual	--	15.9	--	31.7	--	42.3
C, Ca, Al-Si	15.34	96.5	44.78	141.3	38.12	90.1
C, Al-Si, Si	23.53	148.0	28.29	89.2	42.07	99.4
C, Ca, Si	15.38	96.7	32.41	102.2	41.21	99.4
Ca, Al-Si, Si	9.15	57.5	14.98	47.2	74.43	176.0
Average		99.7		95.0		115.7
#4 Actual	--	14.0	--	33.8	--	42.7
C, Ca, Al-Si	10.78	77.0	27.77	82.2	48.79	114.3
C, Al-Si, Si	8.23	58.8	32.70	96.7	47.56	111.4
C, Ca, Si	10.76	76.9	31.62	93.6	47.83	112.0
Ca, Al-Si, Si	12.70	90.7	37.04	109.6	37.49	87.8
Average		75.8		95.9		106.4
#5 Actual	--	12.4	--	34.8	--	43.1
C, Ca, Al-Si	13.56	109.4	50.02	143.7	35.39	82.1
C, Al-Si, Si	22.27	183.1	33.16	95.3	39.59	91.9
C, Ca, Si	13.61	109.7	36.85	105.9	38.67	89.7
Ca, Al-Si, Si	6.98	56.3	18.30	52.6	74.04	171.8
Average		114.6		99.4		108.9
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	98.8	14.6	134.6	30.0	93.4	12.2
C, Al-Si, Si	139.9	48.4	90.0	9.9	101.2	7.3
C, Ca, Si	99.0	14.8	100.1	7.0	99.5	8.2
Ca, Al-Si, Si	68.3	14.3	51.6	39.5	165.1	46.8

Table 24

Computed Constituent Contents - Percent  
River Sand - 3/4 Limestone - Concrete

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. %	Recovery	Pred. %	Recovery	Pred. %	Recovery
#6 Actual	--	21.5	--	26.4	--	42.4
C, Ca, Al-Si	16.29	75.8	29.42	111.6	45.01	106.1
C, Al-Si, Si	19.14	89.0	25.96	98.5	45.43	107.1
C, Ca, Si	16.18	75.3	27.13	103.0	45.28	106.7
Ca, Al-Si, Si	19.46	90.5	26.59	100.9	42.01	99.0
Average		82.6		103.5		104.7
#7 Actual	--	18.2	--	29.6	--	42.7
C, Ca, Al-Si	16.29	89.5	30.85	104.2	45.65	106.9
C, Al-Si, Si	16.29	89.5	20.02	101.4	45.75	107.1
C, Ca, Si	16.26	89.3	30.30	102.4	45.71	107.0
Ca, Al-Si, Si	17.05	93.7	30.17	101.9	44.92	105.2
Average		91.4		102.5		106.6
#8 Actual	--	15.9	--	31.4	--	42.9
C, Ca, Al-Si	23.31	146.6	28.96	92.3	41.81	97.5
C, Al-Si, Si	23.33	146.7	28.93	92.2	41.81	97.5
C, Ca, Si	23.31	146.6	28.94	92.2	41.81	97.5
Ca, Al-Si, Si	23.34	146.8	28.93	92.2	41.78	97.4
Average		146.7		92.2		97.4
#9 Actual	--	14.0	--	33.7	--	42.9
C, Ca, Al-Si	13.16	94.0	35.43	105.1	41.02	95.6
C, Al-Si, Si	12.75	91.1	35.92	106.6	40.96	95.5
C, Ca, Si	13.17	94.1	35.75	106.1	40.98	95.5
Ca, Al-Si, Si	12.71	90.8	35.83	106.3	41.44	96.6
Average		92.5		106.0		95.8
#10 Actual	--	12.4	--	34.8	--	43.1
C, Ca, Al-Si	16.35	131.8	28.18	81.0	37.42	86.8
C, Al-Si, Si	10.15	81.8	35.69	102.6	36.51	84.7
C, Ca, Si	16.59	133.8	33.14	95.2	36.82	85.4
Ca, Al-Si, Si	9.47	76.4	34.32	98.6	43.94	102.0
Average		105.0		94.4		89.7
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	107.5	30.1	98.8	12.2	98.6	8.3
C, Al-Si, Si	100.4	26.2	100.3	5.4	98.4	9.3
C, Ca, Si	107.8	30.7	99.8	5.8	98.4	6.9
Ca, Al-Si, Si	99.6	27.2	100.0	5.2	100.0	3.5

Table 25

Computed Constituent Contents - Percent  
Limestone Sand - 3/4 Limestone

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. %	Recovery	Pred. %	Recovery	Pred. %	Recovery
#11 Actual	--	21.5	--	27.0	--	42.4
C, Ca, Al-Si	9.69	45.1	33.59	124.4	51.22	120.8
C, Al-Si, Si	16.44	76.5	25.93	96.0	56.97	134.4
C, Ca, Si	10.14	47.2	41.47	153.6	45.31	107.0
Ca, Al-Si, Si	119.17	554.3	-55.45	-205.4	-18.02	-42.5
Average		180.7		42.2		79.9
#12 Actual	--	18.2	--	30.0	--	42.8
C, Ca, Al-Si	22.72	124.8	23.49	78.3	42.13	98.4
C, Al-Si, Si	19.40	106.6	27.26	90.9	39.30	91.8
C, Ca, Si	22.50	123.6	19.61	65.4	45.04	105.2
Ca, Al-Si, Si	-31.19	-171.4	67.34	224.5	76.23	178.1
Average		45.9		114.8		118.4
#13 Actual	--	15.9	--	32.2	--	43.0
C, Ca, Al-Si	19.26	121.1	28.30	87.9	42.27	99.3
C, Al-Si, Si	21.03	132.3	26.30	81.7	43.77	101.8
C, Ca, Si	19.38	121.9	30.36	94.3	40.73	94.8
Ca, Al-Si, Si	47.85	300.9	5.05	15.7	24.19	56.3
Average		169.1		69.9		87.8
#14 Actual	--	14.0	--	33.2	--	43.1
C, Ca, Al-Si	17.70	126.4	35.26	106.2	39.56	91.8
C, Al-Si, Si	11.83	84.5	41.91	126.2	34.56	80.2
C, Ca, Si	17.31	123.6	28.41	85.6	44.69	103.7
Ca, Al-Si, Si	-77.40	-552.9	112.61	339.2	99.71	231.4
Average		-54.6		164.3		126.8
#15 Actual	--	12.4	--	335.4	--	43.1
C, Ca, Al-Si	12.07	97.3	36.78	103.9	40.08	93.0
C, Al-Si, Si	13.12	105.8	35.59	100.5	40.97	95.1
C, Ca, Si	12.14	97.9	38.01	107.4	39.16	90.9
Ca, Al-Si, Si	29.10	234.7	22.93	64.8	29.31	68.0
Average		133.9		94.2		86.7
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	102.9	34.4	100.1	17.8	100.5	11.8
C, Al-Si, Si	101.1	21.8	99.1	16.7	100.7	20.4
C, Ca, Si	102.8	33.0	101.3	33.0	92.6	21.2
Ca, Al-Si, Si	73.1	436.2	87.8	208.3	98.3	107.9



Table 26

Computed Constituent Contents - Percent  
Limestone Sand - 3/4 Gravel Concrete

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. %	Recovery	Pred. %	Recovery	Pred. %	Recovery
#16 Actual		21.5		26.6		42.3
C, Ca, Al-Si	25.08	116.5	18.93	71.2	46.10	109.0
C, Al-Si, Si	26.36	122.6	20.47	77.0	42.99	101.6
C, Ca, Si	25.12	116.8	20.07	75.4	43.80	103.6
Ca, Al-Si, Si	20.34	94.6	29.12	109.5	41.36	97.8
Average		112.7		83.3		103.0
#17 Actual		18.2		29.7		42.5
C, Ca, Al-Si	16.03	88.1	31.10	104.7	38.99	91.7
C, Al-Si, Si	12.05	66.2	26.29	88.5	48.67	114.5
C, Ca, Si	15.92	87.5	27.54	92.7	46.15	108.6
Ca, Al-Si, Si	30.78	169.1	-0.57	-1.9	53.75	126.5
Average		102.7		71.0		110.3
#18 Actual		15.9		31.8		42.9
C, Ca, Al-Si	22.89	144.0	21.94	69.0	51.93	121.0
C, Al-Si, Si	30.24	190.2	30.84	97.3	34.03	79.3
C, Ca, Si	23.10	145.3	28.52	89.7	38.68	90.2
Ca, Al-Si, Si	04.37	-27.5	80.46	253.0	24.66	57.5
Average		113.0		127.2		87.0
#19 Actual		14.0		32.9		42.9
C, Ca, Al-Si	10.38	74.1	42.06	127.8	37.96	88.5
C, Al-Si, Si	8.59	61.4	39.90	121.3	42.32	98.6
C, Ca, Si	10.33	73.8	40.46	123.0	41.19	96.0
Ca, Al-Si, Si	17.02	121.6	27.81	84.5	44.60	104.0
Average		82.7		114.2		96.8
#20 Actual		12.4		35.0		42.7
C, Ca, Al-Si	7.76	62.6	41.99	120.0	38.11	89.2
C, Al-Si, Si	4.73	38.2	38.32	109.5	45.49	106.5
C, Ca, Si	7.67	61.8	39.28	112.2	43.57	102.0
Ca, Al-Si, Si	18.99	153.2	17.88	51.1	49.35	115.6
Average		78.9		98.2		103.4
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	97.1	33.1	98.5	27.3	99.9	14.5
C, Al-Si, Si	94.7	61.2	98.7	17.4	100.1	13.1
C, Ca, Si	97.0	33.9	98.6	18.9	100.1	7.1
Ca, Al-Si, Si	102.2	78.0	99.2	95.5	100.3	26.3

Table 27

Constituent Contents - Weight-River  
Sand - 3/4 Gravel Concrete

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery
# 2 Actual		4.8		5.9		9.3
C, Ca, Al-Si	5.69	119.0	8.48	142.8	8.23	88.5
C, Al-Si, Si	7.59	158.8	3.92	66.0	10.12	108.8
C, Ca, Si	5.82	121.8	4.81	81.0	9.75	104.8
Ca, Al-Si, Si	2.25	47.1	04.62	-77.8	26.63	286.3
#3 Actual		4.0		6.6		9.3
C, Ca, Al-Si	3.72	92.3	10.13	152.6	7.54	81.3
C, Al-Si, Si	5.35	132.8	6.24	94.0	9.16	98.8
C, Ca, Si	3.83	95.0	7.00	105.4	8.84	95.4
Ca, Al-Si, Si	0.84	20.8	-0.97	-14.6	23.09	249.1
#4 Actual		3.4		6.9		9.2
C, Ca, Al-Si	3.10	90.1	10.69	155.8	7.44	81.3
C, Al-Si, Si	4.94	143.6	6.28	91.6	9.28	101.4
C, Ca, Si	3.23	93.9	7.14	104.1	8.92	97.5
Ca, Al-Si, Si	-0.16	-4.6	-1.89	-27.6	25.06	273.9
#5 Actual		3.0		7.3		9.2
C, Ca, Al-Si	2.00	66.2	7.04	96.4	10.18	110.4
C, Al-Si, Si	1.95	64.6	7.17	98.2	10.13	109.9
C, Ca, Si	2.00	66.2	7.14	97.8	10.14	110.0
Ca, Al-Si, Si	2.10	69.5	7.42	101.64	9.65	104.7
#6 Actual		2.7		7.6		9.2
C, Ca, Al-Si	2.57	95.5	12.36	163.7	6.48	70.5
C, Al-Si, Si	4.65	172.8	7.36	97.5	8.55	93.0
C, Ca, Si	2.71	100.7	8.34	110.5	8.15	88.7
Ca, Al-Si, Si	-1.13	-72.0	-1.88	-24.4	26.41	287.4
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	92.6	18.37	142.3	26.7	87.4	14.9
C, Al-Si, Si	134.5	41.9	89.5	13.4	102.4	7.1
C, Ca, Si	95.5	19.9	99.8	11.4	99.3	8.3
Ca, Al-Si, Si	18.2	43.6	-8.6	66.3	240.3	77.3

Table 28  
Constituent Contents - Weight-River  
Sand - 3/4 Limestone Concrete

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery
#7 Actual		4.8	5.92	9.5		9.6
C, Ca, Al-Si	3.84	79.5	6.39	107.9	9.96	104.5
C, Al-Si, Si	4.41	91.3	5.76	97.3	10.05	105.5
C, Ca, Si	3.85	79.7	5.98	101.0	10.02	105.1
Ca, Al-Si, Si	4.53	93.8	5.86	99.0	9.39	98.5
#8 Actual		4.1		6.6		9.6
C, Ca, Al-Si	3.87	94.8	6.68	100.6	10.05	104.8
C, Al-Si, Si	3.84	94.1	6.72	101.2	10.04	104.7
C, Ca, Si	3.87	94.8	6.71	101.0	10.04	104.7
Ca, Al-Si, Si	3.83	93.9	6.71	101.0	10.08	105.1
#9 Actual		3.5		7.0		9.6
C, Ca, Al-Si	5.33	150.6	6.54	93.6	9.40	98.3
C, Al-Si, Si	5.54	156.5	6.31	90.3	9.43	98.6
C, Ca, Si	5.34	150.8	6.39	91.4	9.42	98.5
Ca, Al-Si, Si	5.59	157.9	6.35	90.8	9.19	96.1
#10 Actual		3.1		7.5		9.5
C, Ca, Al-Si	2.52	81.0	8.31	111.1	9.14	96.0
C, Al-Si, Si	2.74	88.1	8.06	107.8	9.18	96.4
C, Ca, Si	2.52	81.0	8.15	109.0	9.16	96.2
Ca, Al-Si, Si	2.79	89.7	8.11	108.4	8.90	93.5
#11 Actual		2.8		7.8		9.6
C, Ca, Al-Si	10.94	395.0	0.80	10.3	2.38	24.7
C, Al-Si, Si	3.34	120.6	9.17	117.5	1.15	11.9
C, Ca, Si	10.84	391.3	6.17	79.3	1.59	16.5
Ca, Al-Si, Si	1.82	65.7	7.76	99.7	9.89	102.7
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	160.2	134.4	84.7	42.1	85.7	34.3
C, Al-Si, Si	110.1	29.0	102.8	10.4	83.3	40.1
C, Ca, Si	159.5	132.8	96.3	11.4	84.2	38.0
Ca, Al-Si, Si	100.2	34.3	99.8	6.3	99.2	4.7

Table 29

Constituent Contents - Weight-Limestone  
Sand - 3/4 Limestone Concrete

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery
#12 Actual		4.8		6.0		9.5
C, Ca, Al-Si	2.46	51.0	7.33	121.2	10.99	115.7
C, Al-Si, Si	4.44	92.1	4.99	82.5	12.55	132.1
C, Ca, Si	2.71	56.2	8.75	144.6	10.04	105.7
#13 Actual		4.1		6.8		9.7
C, Ca, Al-Si	4.93	120.0	5.41	79.9	9.61	99.5
C, Al-Si, Si	3.99	97.1	6.51	96.2	8.87	91.8
C, Ca, Si	4.81	117.0	4.74	70.0	10.05	104.0
Ca, Al-Si, Si	-1.60	038.9	10.81	159.6	12.84	132.9
#14 Actual		3.6		7.3		9.8
C, Ca, Al-Si	4.30	119.4	6.40	87.7	9.55	98.0
C, Al-Si, Si	4.64	128.9	6.00	82.2	9.81	100.6
C, Ca, Si	4.34	120.6	6.64	91.0	9.38	96.2
Ca, Al-Si, Si	6.68	185.6	4.43	60.7	8.37	85.8
#15 Actual		3.1		7.4		9.6
C, Ca, Al-Si	4.15	133.0	7.85	105.9	8.98	93.4
C, Al-Si, Si	2.09	67.1	10.29	138.9	7.35	76.4
C, Ca, Si	3.89	124.7	6.37	86.0	9.97	103.6
Ca, Al-Si, Si	-10.30	-330.1	19.82	267.5	16.14	167.8
#16 Actual		2.7		7.8		9.5
C, Ca, Al-Si	2.82	102.9	8.26	105.6	8.97	94.2
C, Al-Si, Si	2.74	100.0	8.35	106.8	8.91	93.6
C, Ca, Si	2.81	102.6	8.20	104.9	9.01	94.6
Ca, Al-Si, Si	2.27	82.8	8.72	111.5	9.25	97.2
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	105.3	32.2	100.1	16.4	100.7	9.0
C, Al-Si, Si	97.0	22.0	101.1	23.3	103.4	16.2
C, Ca, Si	104.2	28.1	99.3	28.2	100.8	5.0
Ca, Al-Si, Si	47.6	252.6	106.1	124.0	105.4	47.3



Table 30  
Constituent Contents - Weight-Limestone  
Sand - 3/4 Gravel Concrete

Signature Combinations	Cement		Fine Aggregate		Coarse Aggregate	
	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery	Pred. (lbs)	Recovery
#17 Actual		4.8		6.0		9.5
C, Ca, Al-Si	5.81	120.0	5.33	89.1	9.06	95.2
C, Al-Si, Si	6.03	124.6	5.50	92.0	8.70	91.4
C, Ca, Si	5.82	120.2	5.42	90.6	8.86	93.1
Ca, Al-Si, Si	4.09	84.5	7.36	123.1	8.89	93.4
#18 Actual		4.0		6.6		9.4
C, Ca, Al-Si	3.46	85.6	6.90	104.6	8.79	93.1
C, Al-Si, Si	1.78	44.1	5.62	85.2	11.51	121.9
C, Ca, Si	3.42	84.6	6.20	93.9	10.28	108.9
Ca, Al-Si, Si	16.58	410.4	-8.55	-129.6	10.05	106.5
#19 Actual		3.5		7.1		9.5
C, Ca, Al-Si	5.23	151.4	5.63	79.9	10.51	110.5
C, Al-Si, Si	8.12	230.7	7.76	110.1	5.99	63.0
C, Ca, Si	5.40	153.4	6.79	96.3	8.05	84.6
Ca, Al-Si, Si	-16.45	-467.3	31.28	443.7	8.42	88.5
#20 Actual		3.1		7.3		9.5
C, Ca, Al-Si	2.11	68.5	8.30	114.5	9.52	100.7
C, Al-Si, Si	1.71	55.5	7.99	110.2	10.17	107.6
C, Ca, Si	2.10	68.2	8.13	112.1	9.87	104.4
Ca, Al-Si, Si	5.24	170.1	4.61	63.6	9.82	103.9
#21 Actual		2.8		7.8		9.5
C, Ca, Al-Si	1.37	49.6	8.23	105.8	9.66	101.8
C, Al-Si, Si	0.33	12.0	7.44	95.6	11.36	119.7
C, Ca, Si	1.34	48.6	7.80	100.3	10.58	111.4
Ca, Al-Si, Si	9.53	345.3	-1.38	-17.7	10.44	110.0
	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$	$\bar{x}$	$S_x$
C, Ca, Al-Si	95.0	40.8	98.8	14.0	100.3	6.8
C, Al-Si, Si	93.4	87.0	98.6	11.2	100.7	24.3
C, Ca, Si	95.0	41.9	98.6	8.3	100.5	11.3
Ca, Al-Si, Si	108.6	347.5	96.6	215.9	100.5	9.1

Table 31

Concrete Tests Series - Cement Contents  
Based on Simple Regression Analysis

Test No.	Actual Content	C, Ca, Al-Si		C, Al-Si, Si		C, Ca, Si		Ca, Al-Si, Si	
		Prediction	Recovery	Prediction	Recovery	Prediction	Recovery	Prediction	Recovery
1a	21.5	9.15	42.6	23.93	111.3	22.04	102.5	19.04	88.6
1b	21.5	22.97	106.8	20.62	95.9	23.48	109.2	21.07	98.0
2a	18.2	5.75	31.6	17.63	96.8	16.83	92.4	13.66	75.0
2b	18.2	10.94	60.1	17.41	95.6	18.48	101.5	15.13	83.1
3a	15.9	6.83	42.9	15.45	97.2	16.09	101.2	12.51	78.7
3b	15.9	5.24	32.9	17.90	112.6	16.29	102.4	13.68	86.0
4a	14.0	2.10	15.0	11.95	85.4	13.26	94.7	8.66	61.9
4b	14.0	18.40	131.4	7.64	54.6	13.45	96.1	10.79	77.0
5a	12.4	-0.59	-4.7	18.96	152.3	16.57	133.0	12.59	101.1
5b	12.4	3.89	31.2	13.51	108.5	13.01	104.5	10.26	82.4
		$\bar{x} = 49.0$		$\bar{x} = 101.0$		$\bar{x} = 103.8$		$\bar{x} = 83.2$	
		$S_x = 41.2 (25.8)$		$S_x = 24.5 (21.6)$		$S_x = 11.4 (9.36)$		$S_x = 11.3 (9.8)$	
River Sand - 3/4 Limestone Concrete									
Test No.	Actual Content	C, Ca, Al-Si		C, Al-Si, Si		C, Ca, Si		Ca, Al-Si, Si	
		Prediction	Recovery	Prediction	Recovery	Prediction	Recovery	Prediction	Recovery
6a	21.5	10.21	47.48	16.77	78.0	20.92	97.3	11.53	53.6
6b	21.5	8.27	38.5	22.41	104.2	18.67	86.8	17.50	81.4
7a	18.2	-22.51	-123.6	14.10	77.4	-14.10	-77.5	8.95	49.2
7b	18.2	37.20	204.4	20.55	112.9	50.26	276.1	14.80	81.3
8a	15.9	119.0	748.0	25.16	158.2	138.60	871.7	18.30	115.1
8b	15.9	-15.75	-99.1	22.40	140.9	-6.9	-43.4	17.30	108.8
9a	14.0	6.37	45.5	14.13	100.9	17.27	123.4	8.25	58.9
9b	14.0	-25.2	180.0	11.54	82.4	-16.80	-120.0	6.02	43.0
10a	12.4	--	--	--	--	--	--	9.55	77.0
10b	12.4	24.9	200.8	5.54	44.7	37.39	301.5	-0.84	-0.7
		$\bar{x} = 138.0$		$\bar{x} = 100.0$		$\bar{x} = 168.0$		$\bar{x} = 66.8$	
		$S_x = 258 (120)$		$S_x = 34.6$		$S_x = 301 (170)$		$S_x = 33.8 (27.9)$	

Table 31 (Cont'd)

Test No.	Actual Content	C, Ca, Al-Si		C, Al-Si, Si		C, Ca, Si		Ca, Al-Si, Si	
		Prediction	Recovery	Prediction	Recovery	Prediction	Recovery	Prediction	Recovery
11a	21.6	41.25	191.8	8.69	40.4	45.41	211.2	26.67	124.0
11b	21.5	34.66	161.2	18.24	89.5	35.45	164.9	27.24	126.7
12a	18.2	8.39	46.1	33.56	184.4	5.05	27.7	19.81	108.8
12b	18.2	13.13	72.1	15.87	86.1	12.72	69.9	14.36	78.9
13a	19.98	9.98	62.8	23.83	149.9	7.57	47.6	15.91	100.1
13b	15.9	24.37	153.3	30.58	2204.9	23.23	146.1	28.15	177.1
14a	14.0	7.73	55.2	17.87	127.7	6.35	45.3	12.40	88.6
14b	14.0	32.50	232.2	-5.27	-37.3	37.36	266.8	15.59	111.3
15a	12.4	14.57	117.5	35.70	287.9	11.76	94.8	24.17	194.9
15b	12.4	56.35	453.7	-18.10	-146.0	50.94	410.8	15.60	125.8

$\bar{x} = 154.6$   
 $S_x = 122.6 (85.2)$

$\bar{x} = 98.8$   
 $S_x = 124.7 (55.4)$

$\bar{x} = 148.5$   
 $S_x = 120.9 (80.2)$

$\bar{x} = 123.6$   
 $S_x = 36.7 (27.5)$

Limestone Sand = 3/4 Gravel Concrete

Test No.	Actual Content	C, Ca, Al-Si		C, Al-Si, Si		C, Ca, Si		Ca, Al-Si, Si	
		Prediction	Recovery	Prediction	Recovery	Prediction	Recovery	Prediction	Recovery
16a	21.5	18.06	84.0	22.29	103.6	18.98	88.3	7.40	34.4
16b	21.5	23.60	108.8	20.76	96.6	22.72	105.7	31.83	147.9
17a	18.2	15.85	87.1	7.17	39.4	13.71	75.3	37.39	205.4
17b	18.2	16.18	88.9	12.35	67.8	16.28	83.9	24.64	135.4
18a	15.9	21.71	136.5	24.14	151.8	22.17	139.4	15.82	99.5
18b	15.9	16.59	104.3	34.08	214.3	20.86	131.2	-29.48	-185.4
19a	14.0	15.42	110.1	8.18	58.4	13.43	95.9	35.39	252.8
19b	14.0	12.00	85.7	17.41	124.4	13.22	94.4	-1.81	-12.9
20a	12.4	7.03	56.7	3.84	31.0	6.06	48.9	16.17	130.4
20b	12.4	15.24	122.9	13.43	108.3	14.62	117.9	20.71	167.0

$\bar{x} = 98.6$   
 $S_x = 22.8 (12.9)$

$\bar{x} = 99.6$   
 $S_x = 55.4 (50.1)$

$\bar{x} = 98.1$   
 $S_x = 26.9 (22.1)$

$\bar{x} = 97.4$   
 $S_x = 125.5 (102.1)$

Tables 23 through 26 clearly indicate that the accuracy of the computed cement contents is dependent on the three signatures used in the computation. The river sand 3/4-gravel series (Table 23) had mean recoveries for cement content of about 99 percent and associated standard deviations of about 15 percent for the C, Ca, and Al-Si signature combination and for the C, Ca, and Si combination. But for the Ca, Al-Si, and combination, mean cement recoveries decreased to 68 percent and standard deviations remained at 14.3 percent. The C, Al-Si, and Si combination produced even greater errors, with a mean cement recovery of about 140 percent and a standard deviation of 48.4 percent.

The computational results from the other three concrete test series (Tables 24, 25, and 26) exhibited somewhat similar trends. The river sand 3/4-limestone computations had mean cement recoveries ranging from about 108 percent for the C, Ca, and Si combination to nearly 100 percent for the Ca, Al-Si, and Si combination. The associated standard deviations were 30.7 and 26.2 percent for the C, Ca, and Si combination and the C, Al-Si, and Si combination, respectively. The limestone sand-3/4 limestone (Table 25) had standard deviations for cement recovery ranging from 21.8 percent (C, Al-Si, and Si) to 436 percent (Ca, Al-Si, and Si). Similarly, the limestone sand-3/4 gravel (Table 26) had standard deviations for cement recovery ranging from about 33 percent (C, Ca, and Al-Si) to 78 percent (Ca, Al-Si, and Si). An evaluation of the various signature combinations indicates that overall, no consistent set of three signatures is more accurate than the others. Although there are certain key signatures that were normally present in the most accurate computations, the most predominant signature is C; in most cases, Ca is another predominant signature.

In many cases, the computed fine and coarse aggregate contents were more accurate than the computed cement contents (Tables 23 through 26). If the aggregate computations were consistently more accurate, they and the water contents could be used to compute cement content, since the constituents contents of the concrete always add up to unity. For this approach to work, however, the level of accuracy difference must be significant, and a quick check of results indicates that the difference is neither significant nor consistent.

The results of the weight computations (Tables 27 through 30) were similar to those of the percent computations, with the exception that the standard deviations for cement and aggregate recoveries were normally greater than those obtained from the percent computations. For example, the lowest standard deviation (cement recovery) for the weight computed results was 19 percent, and at least half of the cement recovery standard deviations were greater than 40 percent.

The cement contents from the multipliers derived from the simple linear regression analysis (Table 31) were computed for each neutron/gamma test (two tests per mix). As with the previous computations, means and standard deviations were computed for each concrete test



series. The standard deviations in parentheses were computed from the average cement contents for each mix. The standard deviations of the average cement contents should be used when comparing with the accuracy of the other two sets of computed constituent contents (Tables 23 through 30). A direct comparison of standard deviations will indicate that the simple linear regression multipliers for the C, Ca, and Si river sand 3/4 gravel series produced the most accurate results of all the multipliers, a 9.4 percent standard deviation. Also the C, Ca, and Al-Si simple linear regression multipliers for the limestone sand-3/4 gravel test series were the most accurate multipliers for that test series. Conversely, more than half (nine) of the standard deviations from the simple linear regression multipliers were greater than 40 percent. Thus, on the average, the simple linear regression multipliers were slightly less accurate than the multiple linear regression multipliers percent.

A comparison of the standard deviations in parentheses (Table 31) and those based on the individual neutron/gamma tests indicates that averaging of neutron/gamma tests per batch increases the reliability of the answer. It can also be presumed that increasing the number of neutron/gamma tests per batch to more than two would further increase the accuracy of predicting cement content. But since each neutron/gamma test requires 11 min to run, repetitive testing of the same batch or sample decreases system rapidity so that it is no longer a major advantage of its use.

Two general phenomena have been observed for all sets of multipliers used to compute cement content: (1) the river sand 3/4 gravel concrete test series produced the most consistent results; and (2) no given set of three multipliers produced the most accurate results for all four concrete test series. These phenomena indicate that aggregate chemical composition influences the accuracy of the cement prediction. The aggregate influence is assumed to be related to the quantity of Ca in the aggregates. Furthermore, as aggregate chemical composition changes, the signatures producing the most accurate results will change.

#### Kelly-Vail Results

The data obtained from the Kelly-Vail water and cement content tests were analyzed to determine overall accuracy and the influence of aggregate type on test results. As with the neutron/gamma tests, percent recovery was the basis of comparison. The water tests were related to both the free and total water content of the mixes.

Table 32 indicates that for all samples, the average recovery for free water was 101.2 percent, and for total water, 88.8 percent. The associated standard deviations on a per test basis were 5.14 percent for free water and 6.97 percent for total water. When the results are

broken down and computed on a test series basis, mean free water recoveries varied from 98.1 to 102.9 percent, and mean total water recoveries varied from 83.3 to 93.6 percent. The associated accuracies for the individual test series varied from about 3 to 6 percent.

Table 32  
Summary of Kelly-Vail Test Results

	Water Recovery		Cement Recovery	
	Total Free %	Free %	W/o Agg Blk %	With Agg Blk %
River Sand $\bar{x}$	83.3	102.1	97.8	89.7
3/4 Gravel $S_x$	3.4	4.2	4.0	2.4
$n^* = 10$				
River Sand $\bar{x}$	93.0	102.9	99.5	88.9
3/4 Limestone $S_x$	5.5	6.1	3.4	3.1
$n = 10$				
Limestone $\bar{x}$	93.6	101.7	125.6	96.8
3/4 Limestone $S_x$	5.1	5.7	15.9	10.7
$n = 10$				
Limestone Sand $\bar{x}$	83.5	98.1	118.4	92.5
3/4 Gravel $S_x$	3.0	3.7	8.5	6.7
$n = 10$				
All Data $\bar{x}$	88.8	101.2	110.3	92.0
Combined $S_x$	6.2	5.2	15.1	7.1
$n = 40$				

\*  $n$  = number of samples.

The cement contents were computed directly from the cement content test data and from the cement data after the cement equivalence of the aggregates (aggregate blanks) was subtracted. Table 32 indicates that for all samples, the average recovery for cement content without the aggregate blanks was 110.3 percent, and with the aggregate blanks 92.0 percent. The associated standard deviations (per test) were 15.1 percent for cement contents without aggregate blanks and 7.1 percent for cement contents with aggregate blanks. The means and standard deviations were also computed for each test series. Evaluating the individual test series results illustrates the influence that limestone sand has on the measured cement contents. The cement recovery without

aggregate blanks averaged 125.6 and 118.4 percent for the two limestone sand test series. The equivalent results for the two river sand test series were 99.5 and 97.8 percent.

The above phenomenon in the Kelly-Vail cement tests can readily be understood when it is realized that the cement contents are based on the quantity of Ca passing the No. 50 sieve above the washing machines. All cement and some of the aggregate pass this sieve; if this aggregate contains Ca, the Ca will read out as if it were cement. Obviously, limestone sands contain significant quantities of Ca that will pass the No. 50 sieve (Tables 11 and 12). For example, where the Kelly-Vail cement content test was run on a 325-g sample of limestone sand, the cement equivalency of the limestone sand was 3.8 percent cement. The cement equivalency for the same weight of river sand was 0.9 percent. The cement equivalency of 430 g of the 3/4 in. (1.90 cm) limestone and gravel was 0.8 and 0.4 percent, respectively. Thus, even the dust from the coarser aggregates can contribute to the measured cement contents. But it is encouraging to note that when the cement equivalency blanks were used, the resulting accuracy for all samples was 7.1 percent for cement content.

#### *Strength Predictions*

The neutron/gamma and Kelly-Vail estimates of water and cement contents were used with air content (air meter) to estimate the 28-day compressive strengths of the concrete mixes. The series of curves in Figure 26\* were used to relate the water, cement, and air contents to 28-day compressive strengths. Kelly-Vail strength estimates were obtained for cement contents with and without aggregate blanks removed. The neutron/gamma strength estimates were computed by subtracting the absorption capacities of the aggregates from the neutron/gamma water estimates. The resulting water content after the subtraction presumably represents the free water in the concrete mixes. (Free water is a better estimate of strength than total water.<sup>11</sup>) The Kelly-Vail water contents are more closely related to free water than to total water contents.

Table 33 provides the neutron/gamma and Kelly-Vail strength estimates as well as the actual 28-day cylinder strengths for each of the 20 concrete mixes.

\* Developed from regression analysis of Kelly-Vail data presented in P. A. Howdysshell, "Correlating Kelly-Vail Test Results to the Strength Potential of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (CERL, May 1975).

<sup>11</sup> T. C. Powers, "The Physical Structures and Engineering Properties of Concrete," *Bulletin 90* (Research and Development Laboratories of the PCA, July 1958).

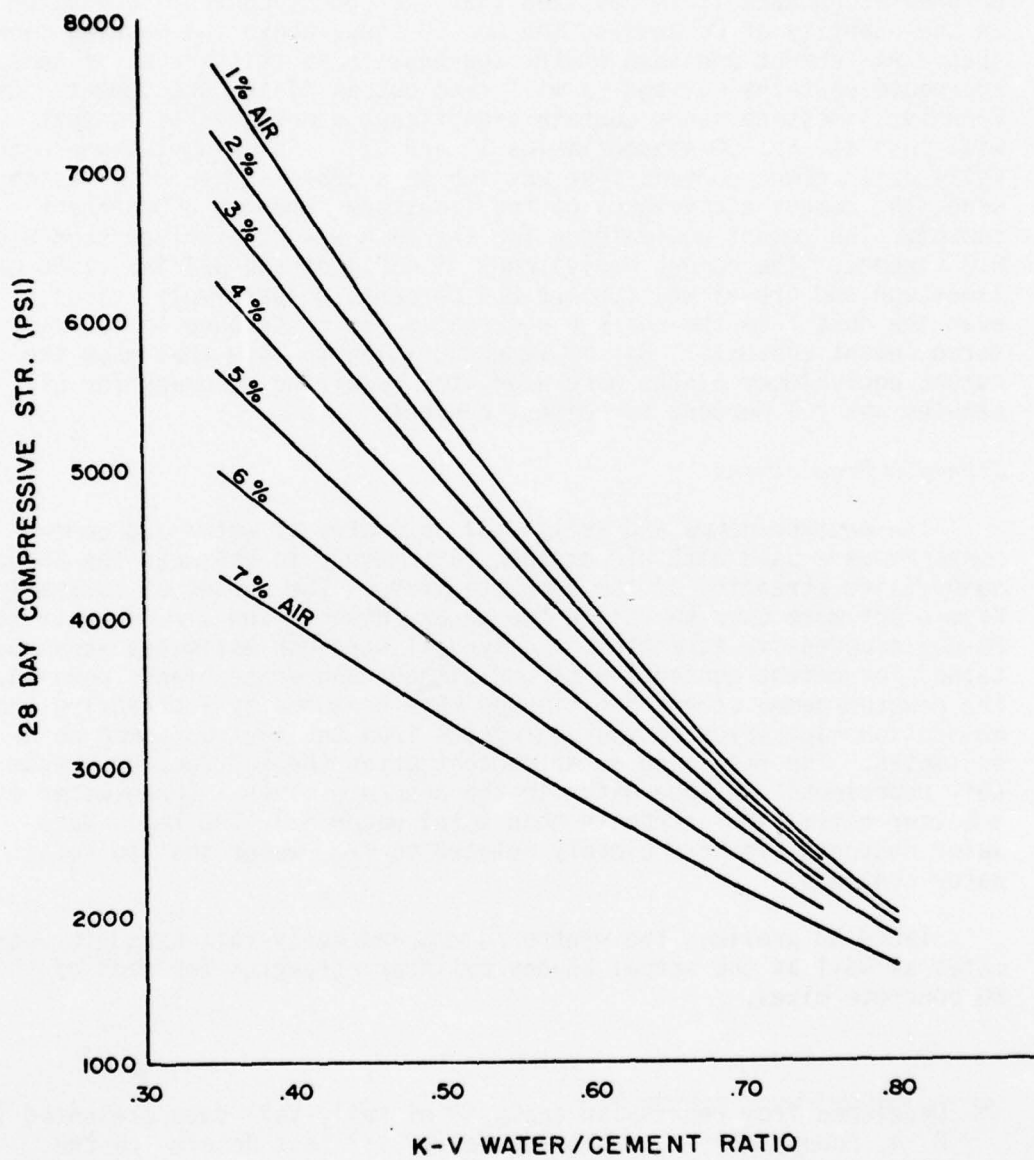


Figure 26. Strength vs. water/cement and air.



Kelly-Vail and Neutron/Gamma Strength Estimates

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Table 33 (Cont'd)

Mix No.	Actual Str (Avg) PSI (28 Day)	N/G Str. Est. (PSI)	Difference N/G. - Act.	K/V w/o Blk Stress (PSI)	Difference K/V w/o - Act.	K/V w Blk Str. Est. (PSI)	Difference K/V w - Act	Sand Blk	Difference Sand blk - Act.
<u>Limestone Sand - 3/4 Limestone</u>									
11	6340	4450	-1890	7200	+860	6100	-240	6300	-40
		6050	-290	7400	+1060	6400	+60	6650	+310
12	6550	6800	+250	7200	+650	5800	-750	6100	-450
		5300	-1250	6800	+250	5300	-1250	5650	-900
13	5240	5650	+410	6300	+1060	4400	-840	4800	-440
		7700	+2460	6800	+1560	5100	-140	5500	+260
14	4440	5000	+560	5450	+1010	3100	-1340	3600	-840
		--	--	5150	+710	2700	-1740	3200	-1240
15	4100	7650	+3550	6000	1900	4000	-100	4250	+150
		--	--	5200	1100	2800	-1300	3200	-900
									$S_x = 797$
<u>Limestone Sand - 3/4 Gravel</u>									
16	6070	5250	-820	7700	+1630	7000	+930	7100	+1070
		7050	+980	7300	+1230	6500	+430	6600	+530
17	5770	4500	-1270	6050	+280	4700	-1070	4850	-920
		5200	-570	7000	+1230	6000	+230	6150	+380
18	4730	6300	+1570	5900	+1170	4350	-380	4500	-230
		5050	+320	5850	+1120	4100	-630	4350	-350
19	4130	4000	-130	5100	+970	3000	-1130	3300	-830
		2600	-1530	5400	+1270	3400	-730	3700	-430
20	3640	--	--	4450	+810	2000	-1640	2450	-1210
		4600	+960	4950	+1310	2700	-940	2950	-690
									$S_x = 879$
									$S_x = 1080$
									$S_x = 1379$
									$S_x = 1253$
									$S_x = 1161$

The accuracy of the strength predictions was determined by the following equation:

$$S_x^2 = \frac{\sum d_i^2}{n-3} \quad [\text{Eq 3}]$$

where  $S_x$  = standard deviation

$d_i$  = difference between the actual and estimated strength of the  $i$ th case

$n$  = sample population

The standard deviations were computed on a test series basis for each of the Kelly-Vail and the neutron/gamma strength predictions.

The standard deviations for the Kelly-Vail strength predictions varied from 411 to 1389 psi, depending on the test series (aggregates) and whether the aggregate blanks were subtracted. The most accurate predictions occurred for the two river sand test series when the aggregate blanks were not used. Standard deviations of 411 and 793 psi were obtained for the river sand 3/4 gravel and river sand 3/4 limestone, respectively. The two limestone sand series were only slightly less accurate when only the sand blanks were used. The standard deviations were 797 and 879 psi for the limestone sand 3/4 limestone and limestone sand 3/4 gravel test series, respectively.

The Kelly-Vail strength estimates computed for the aggregate blanks removed (both coarse and fine) were less accurate than the above estimates. This is probably a result of the prediction curves (Figure 26) being based on only concretes with siliceous sands and without the aggregate blanks removed.

The neutron/gamma strength estimates were considerably less accurate than the Kelly-Vail estimates, but the two test series containing the 3/4 gravel produced usable results. The river sand 3/4 gravel and the limestone sand 3/4 gravel accuracies were 868 and 1253 psi, respectively. The two 3/4 limestone test series had standard deviations of 2229 and 2207 psi.

It should also be noted that since the strength prediction curves (Figure 26) were based on previous Kelly-Vail data, their use biases the accuracy of the neutron/gamma strength estimates; however, a review of the results (Table 33) indicates that the neutron/gamma estimate ranged from very low to very high, indicating that the regression curves were a reasonable fit for the neutron/gamma data.

The resulting accuracies of both the Kelly-Vail and neutron/gamma strength predictions indicate that there is a direct relationship

between the accuracy of the water and cement content determinations and the accuracy of the strength predictions. This relationship is particularly noticeable for the neutron/gamma data.



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A COMPARATIVE EVALUATION OF THE NEUTRON/GAMMA AND KELLY-VAIL TE--ETC(U)  
MAY 77 P A HOWDYSELL

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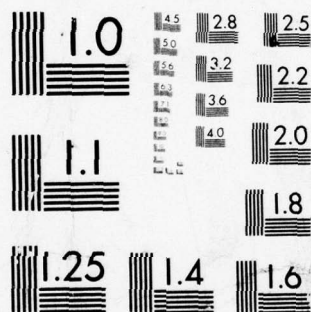
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## 6 NEUTRON/GAMMA TECHNIQUE VERSUS KELLY-VAIL TECHNIQUE

When comparing the neutron/gamma technique to the Kelly-Vail technique for fresh concrete analysis, the following criteria are used:<sup>12</sup>

1. Cement and water contents should have an accuracy of approximately 1 percent and an adequate reproducibility.
2. No prior detailed knowledge of the aggregate or cement properties should be required.
3. The technique should be suitable for use with a wide range of aggregate and cement types.
4. The technique should be capable of being carried out rapidly, preferably within 10 min.
5. It should be operable by trained, but not necessarily highly qualified personnel.
6. It should be suitable for use in the field and acceptable to engineers.
7. The separated mixture-constituents should be available for inspection.
8. The technique should use robust apparatus capable of automation.
9. The technique should be relatively inexpensive in terms of manpower, apparatus, and materials requirements.

A review of the data indicates that the neutron/gamma and Kelly-Vail water content accuracies (standard deviations from recovery) were about the same: Kelly-Vail - 5.2 percent, and neutron/gamma - 6.4 percent; however, the Kelly-Vail cement prediction accuracies were considerably better than those of the neutron/gamma: Kelly-Vail was 7.1 percent overall, and neutron/gamma ranged from 9 to 22 percent for each test series.

The calibration of the neutron/gamma system is an order of magnitude greater than the calibration of the Kelly-Vail system. The Kelly-Vail system requires only that a cement blank and an aggregate blank

<sup>12</sup> R. A. Kenny and B.M.L.G. Tullock, "Analysing Fresh Concrete," *Concrete*, Vol 6, No. 3 (March 1972), pp 23-27.

be run for cement content, and a chloride blank for water content. The total calibration time for the Kelly-Vail system is less than 20 min. Since the neutron/gamma system was not able to obtain the constituent multipliers from tests on the dry constituents, the calibration of the neutron/gamma system would require the running of a concrete mix series in which each constituent (cement, water, fine aggregate, and coarse aggregate) proportions are varied. The time required to do this would involve several days and a minimum of two personnel.

Theoretically, the neutron/gamma system should be suited for use with a wider range of aggregate and cement types than the Kelly-Vail system. The accuracy of the Kelly-Vail cement test is related to the quantity and variability of the fine calcareous aggregates present; however, even in the worst case, in which a calcareous sand is used, the accuracy of the Kelly-Vail system is equal to or greater than the accuracy of the neutron/gamma cement estimate.

Both the neutron/gamma and Kelly-Vail tests can be conducted reasonably quickly. The total irradiation and counting time for the neutron/gamma test is 11 min. If computerized data reduction equipment is used, the net peaks and related water and cement contents can be obtained almost instantaneously after completion of the irradiation time. The Kelly-Vail water and cement content tests require 7 to 8 min each; water and cement content results are produced directly from graphs relating test results to water and cement contents. It should be noted that the Kelly-Vail test requires a 15- to 20-min clean-up time before a second test can be initiated. No clean-up time for the neutron/gamma tests is required if a spare set of sample containers is available.

Operationally, both systems are sufficiently simple and can be operated by technician-level staff; however, the neutron/gamma system is the simpler system, and requires less operator time than the Kelly-Vail system.

The Kelly-Vail system has been proved to be field operational.<sup>13</sup> The neutron/gamma system has never been field-tested, but there is no indication that it could not be used in the field.

The Kelly-Vail system allows for inspection of the aggregate constituents retained on the No. 4 and No. 50 sieves. The neutron/gamma test does not involve separation, so constituents are not available for inspection.

<sup>13</sup> P. A. Howdysshell, "Concrete Quality Control 28 Days-24 Hours-15 Minutes," ACI International Symposium on Accelerated Strength Testing (October 1976).



The Kelly-Vail system can operate in the field with a minimum amount of downtime.<sup>14</sup> The robustness of the neutron/gamma system is unknown, although the NaI crystals are known to be sensitive and capable of being damaged by significant temperature fluctuations.

Without a detailed analysis, it is assumed that the operating costs of the two systems are similar. A previous study has indicated that each Kelly-Vail test costs approximately \$15.<sup>15</sup> However, the capital costs of the two systems' equipment differs significantly. The Kelly-Vail equipment costs between \$4000 and \$6000; the neutron/gamma equipment in its present form costs approximately \$50,000.

<sup>14</sup> P. A. Howdyshell, "Concrete Quality Control 28 Days-24 Hours-15 Minutes."

<sup>15</sup> P. A. Howdyshell, *Operations Guide--Water and Cement Content of Fresh Concrete*, Technical Report M-177/ADA022697 (CERL, September 1975).

## 7 CONCLUSIONS AND RECOMMENDATIONS

### Conclusions

The following major conclusions can be drawn from this study:

1. The Kelly-Vail technique is more accurate than the neutron/gamma technique in the determination of water and cement contents of fresh concrete.

<u>Technique</u>	<u>Accuracy--Water</u>	<u>Accuracy--Cement</u>
Kelly-Vail	5 percent	7 percent
neutron/gamma	6 percent	9 to 22 percent

2. The Kelly-Vail system is a better estimator of concrete strength potential than the neutron/gamma system. Standard deviations for Kelly-Vail strength estimates varied from 411 to 344 psi, depending on aggregate type (calcareous and siliceous). Standard deviations for the neutron/gamma system varied from 868 to 2229 psi.

3. Dry constituent tests cannot be directly used to calibrate the neutron/gamma system; this system requires running a concrete mix series in which each constituent (cement, water, fine aggregates, and coarse aggregates) proportion is varied.

### Recommendations

Noting the above conclusions, and realizing that the cost of the neutron/gamma equipment is 10 times that of the Kelly-Vail equipment, it is recommended that further evaluation of the neutron/gamma system be discontinued until major technological changes occur.

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## APPENDIX:

### ANALYSIS AND DISCUSSION OF TEST RESULTS

#### Analysis of Signature-Constituent Responses

The objective of the signature-constituent analysis is to determine: (1) the linearity between signature intensities and the associated elemental intensities of the test samples; (2) the sensitivity of the signatures to other elements (matrix effects) in the test samples; (3) the relationship between the signatures for individual constituent samples (cement, fine aggregates, and coarse aggregates) and their associated signatures when combined and in the presence of water; and (4) the theoretical and operational accuracy of each signature.

#### *Hydrogen Signature*

The results contained in the neutron/gamma paper indicated that the H signature had a theoretical accuracy of 0.5 to 2.0 percent, and that the H signature was reasonably linearly related to the water content of the sample (Figure A1). The paper also indicated that for the six-channel peak width used, the three background channels on the left side appeared to override a near by 1.94 MeV Ca peak. Thus, variations in the Ca content of the sample influenced the background count of the H signature. The three mortar test series presented in Figure A1 clearly indicate that increasing Ca contents decreases the net H signature.<sup>17</sup>

The Ca interference problem was evaluated in the mortar test series. The TNC spectra for approximately 10 channels on either side of the 2.22 MeV H peak were plotted for three cement and three ottawa sand constituent tests (Figure A2). The spectra from the dry cement and cement paste tests have a major peak (1.94 MeV, Ca full escape peak) seven channels to the left of the H peak. The dry ottawa sand and ottawa sand-water samples do not exhibit this peak. Ottawa sand does not contain Ca, and cement is approximately 45 percent Ca. It is apparent that for the six-channel peak width, the three background channels on the left side are ascending the neighboring Ca peak. It is also apparent that reducing the width of the peak band to four

<sup>16</sup> P. A. Howdyshell, "Preliminary Evaluation of the Neutron/Gamma Technique to Determine the Water and Cement Content of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (CERL, May 1975).

<sup>17</sup> Howdyshell, "Preliminary Evaluation of the Neutron/Gamma Technique."

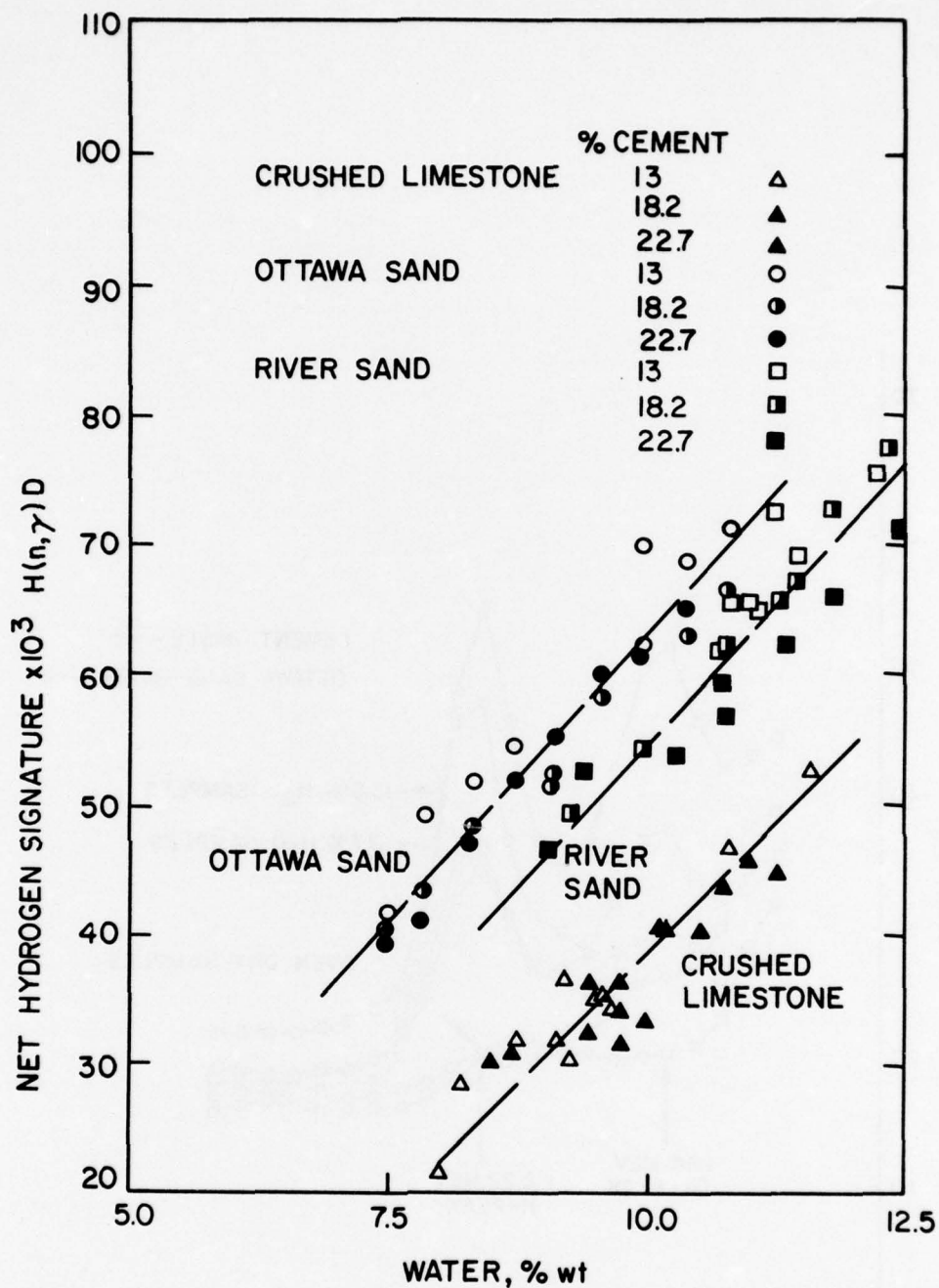


Figure A1. Water content vs. hydrogen signature (TNC). (From P. A. Howdyshe, "Preliminary Evaluation of the Neutron/Gamma Technique to Determine the Water and Cement Content of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 [CERL, May 1975]).

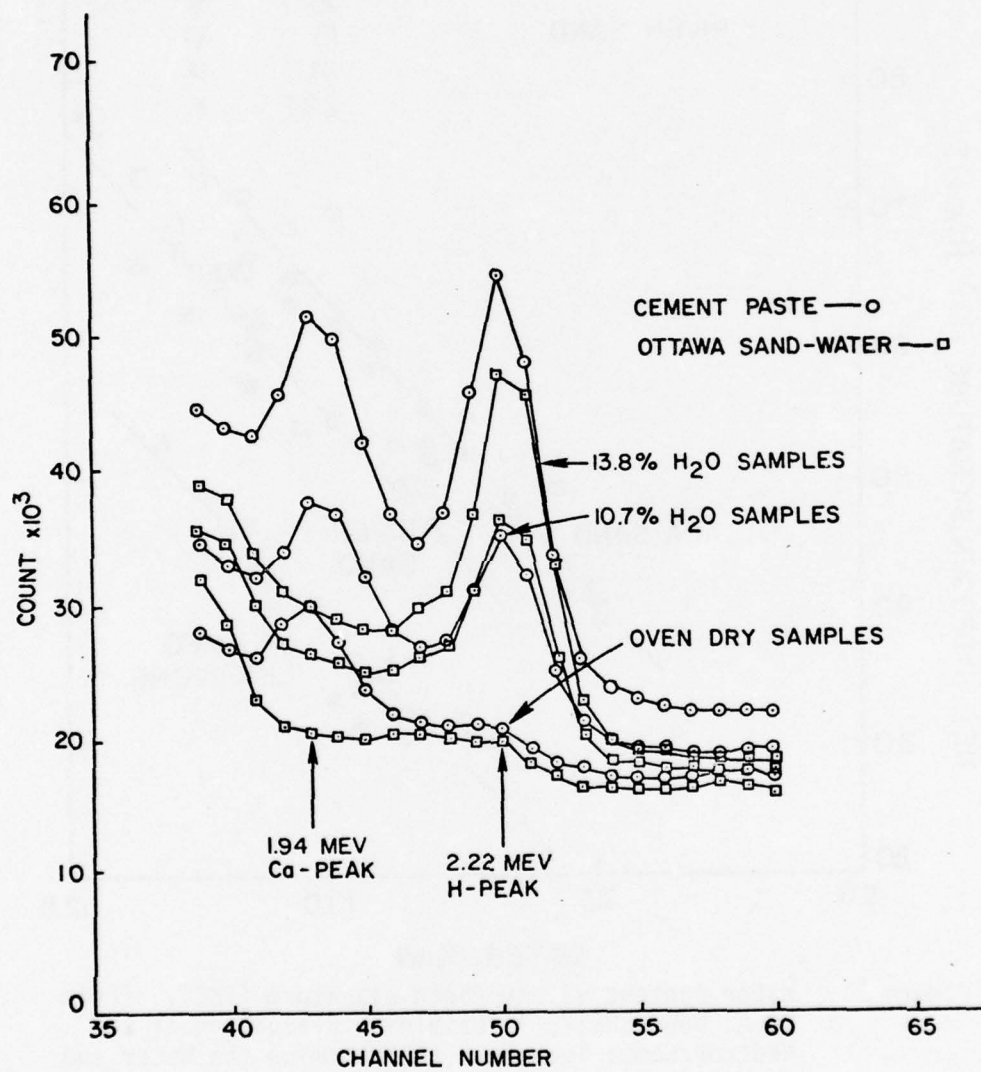


Figure A2. TNC spectra cement paste/ottawa sand-water.



channels should decrease the significance of the Ca interference. Similar observations and conclusions can be drawn from the TNC spectra on the concrete test samples (Figures 12, 13, 14, and 15).

Both four- and six-channel net peaks were computed for the ottawa sand-mortar tests and their associated constituent tests (ottawa sand-water and cement paste). Since it is assumed that the H signature is unique and directly related to water content, the net H peaks were plotted relative to the water content of each mix. Water contents were computed both on a weight basis and a percent basis. Sample density is the difference between the two water content values. Linear regression curves and related coefficients of determinations were computed for the ottawa sand-water and cement paste data. Figures A3 and A4 are the six-channel plots for water content percent and weight, respectively. Using the coefficient of determination  $r^2$  to evaluate how well the data fit the linear regressions (at  $r^2 = 0$  there is no fit and at  $r^2 = 1.0$  there is a perfect fit), it is noted that the best fit occurs for the plots of H signature versus water content-weight. Figures A5 and A6 are the four-channel plots for water content percent and weight, respectively. As with the six-channel plots, the best regression fits occur for the H signature versus water content weight plots. These findings tend to indicate that for the variation in sample density tested, the H signature is related more to the quantity of H (water) present in the sample than it is to the relative percentage of H present in the sample; however, it should be noted that the level of fit for both relationships was very good.

Linear regression curves were computed for the combined ottawa sand-water and cement paste data in Figures A3 through A6. Using coefficients of determination to evaluate regression fits, it is apparent that for the combined data, the best fits occur for the four-channel signatures; it is also apparent that of the two four-channel relationships, the water content-weight data produced the best fit (Figure A5 versus Figure A6). Since ottawa sand does not contain Ca and cement contains approximately 45 percent Ca, the degree of fit for the combined data indicates that the four-channel H signature is relatively insensitive to the quantity of Ca in the sample.

Four-channel net peaks were used to compute the H signatures for all the remaining tests. Figure A7 illustrates the H signature versus the water content weight data for the river and limestone sand mortars and their associated constituent-water tests. Linear regression curves were computed for each of the three constituents and for all constituents combined. The coefficient of determination for the combined data is very close to that obtained for the ottawa sand and cement data--0.973 and 0.978, respectively.

Figure A8 is the H signature (rate intensity) versus water content-percent data for the concrete constituent tests. Figure A9 is

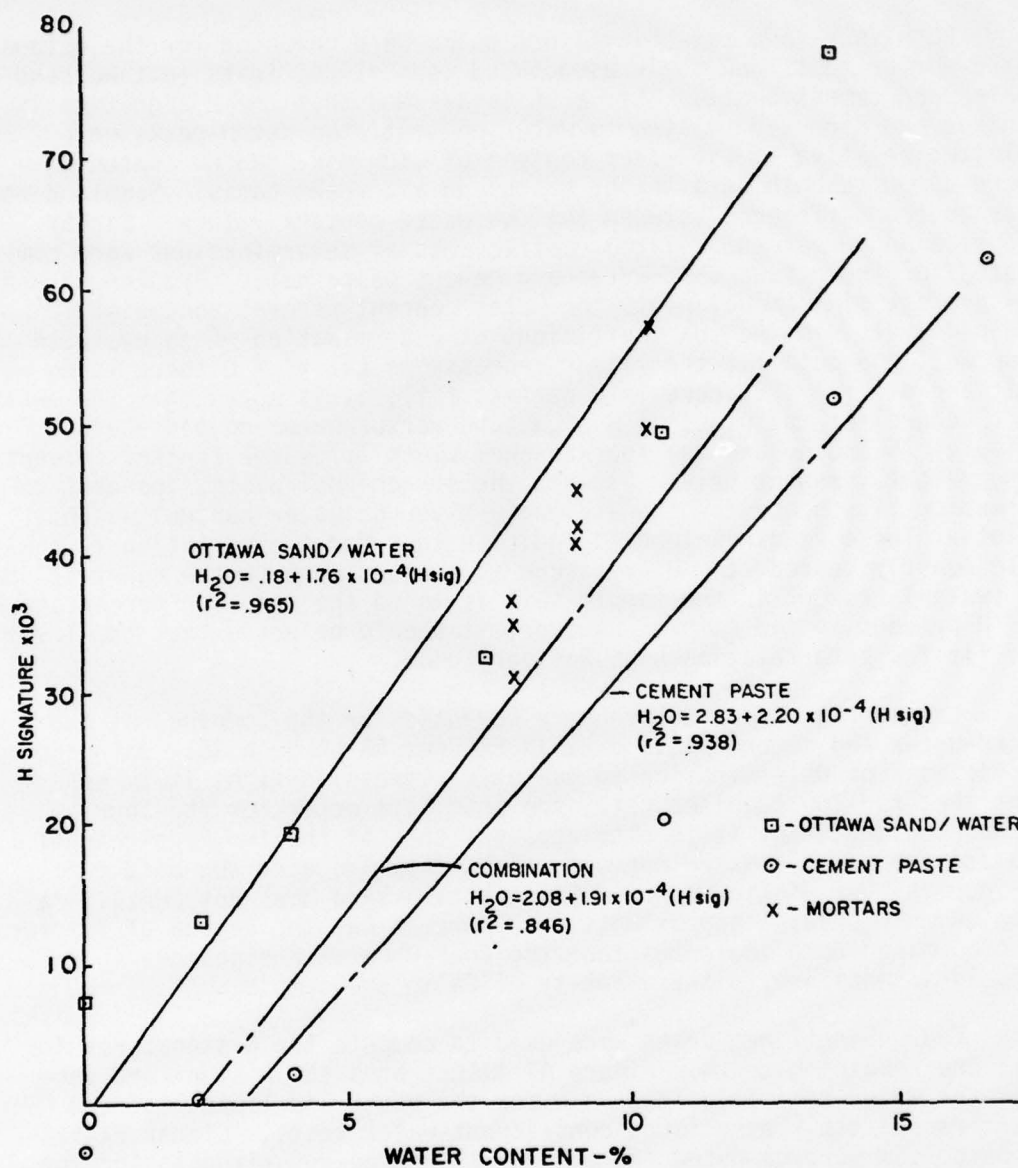


Figure A3. Water content vs. hydrogen signature six-channel hydrogen peak--water content in percent.

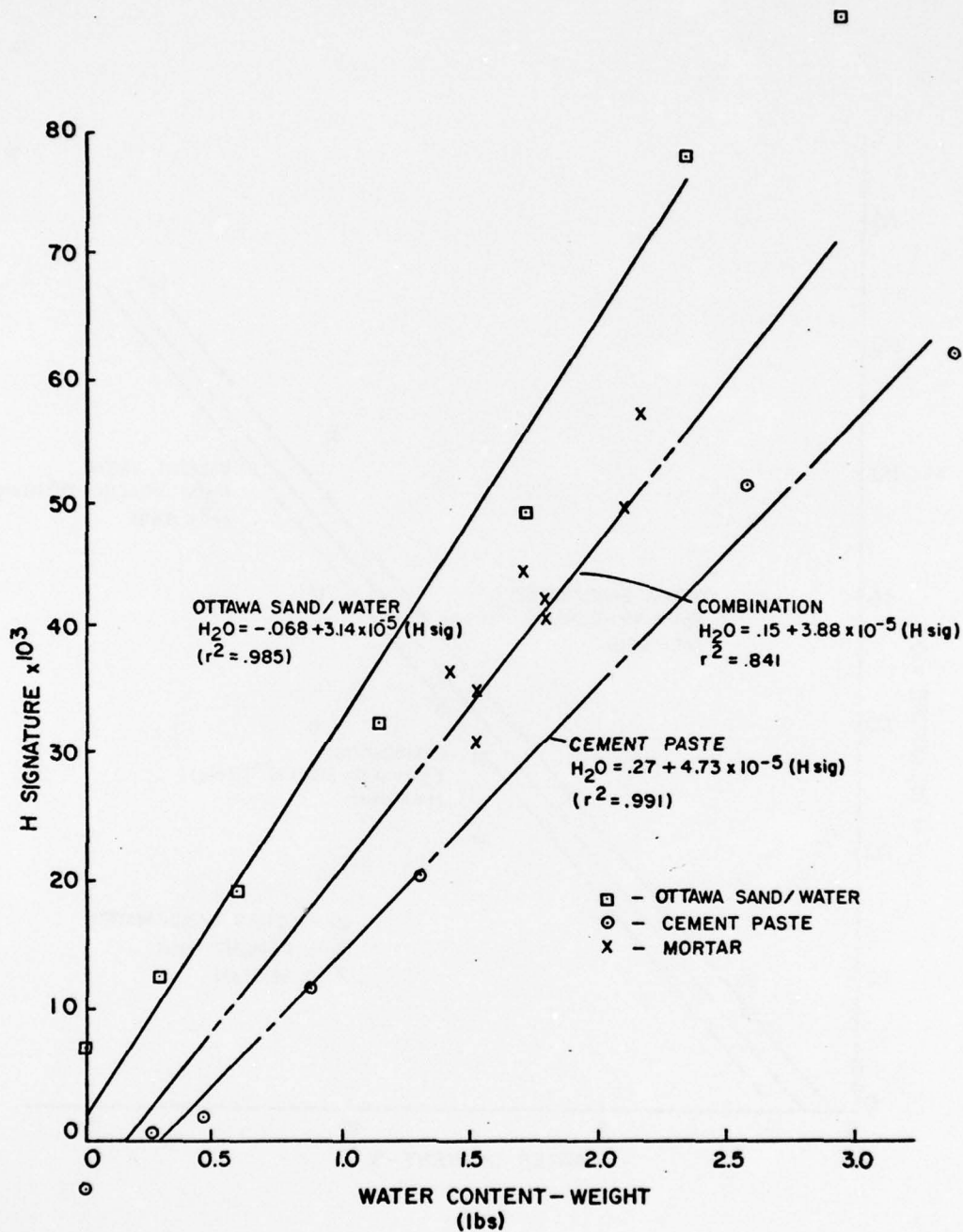


Figure A4. Water content vs. hydrogen signature six-channel hydrogen peak--water content in weight.

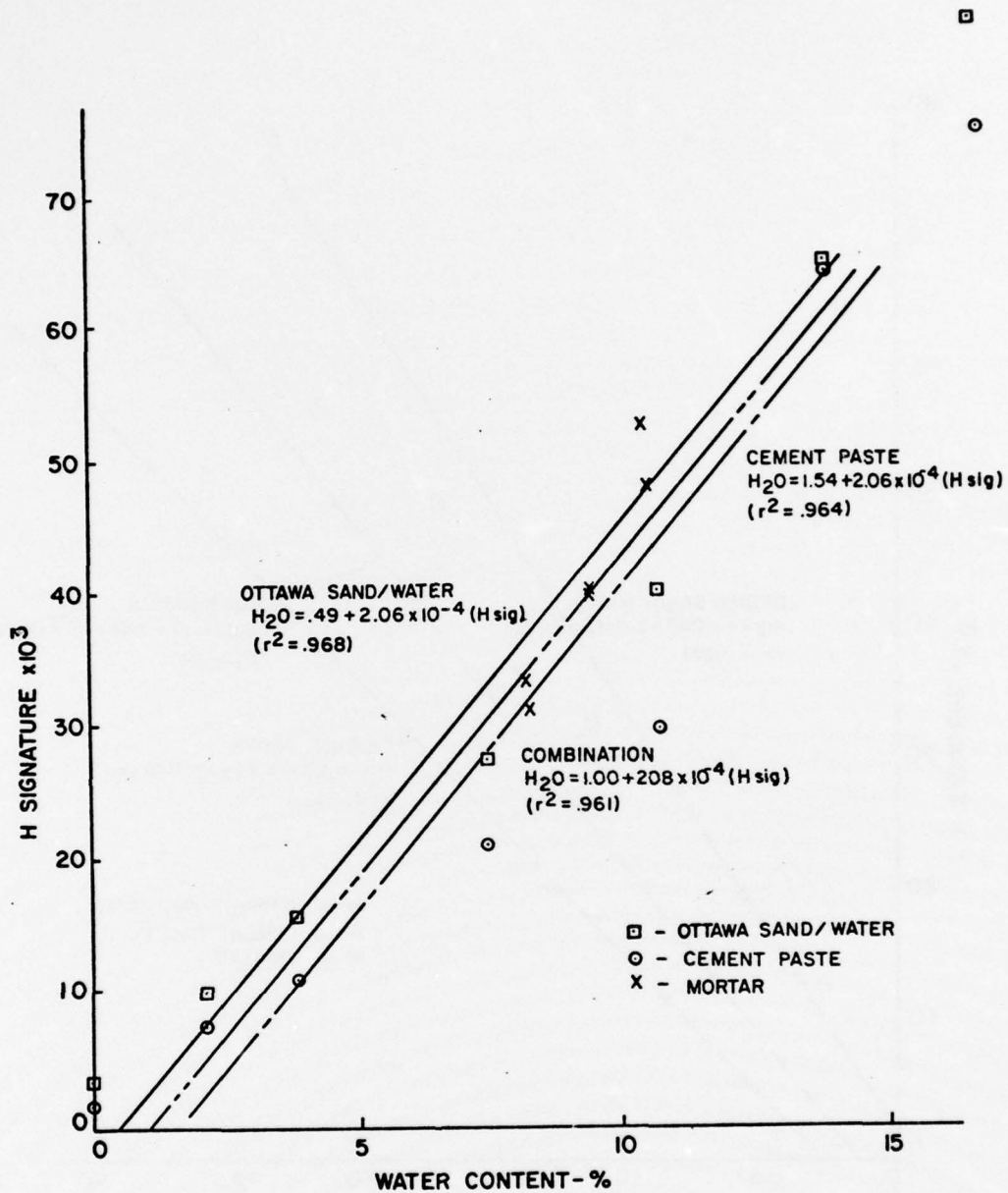


Figure A5. Water content vs. hydrogen signature four-channel hydrogen peak--water content in percent.



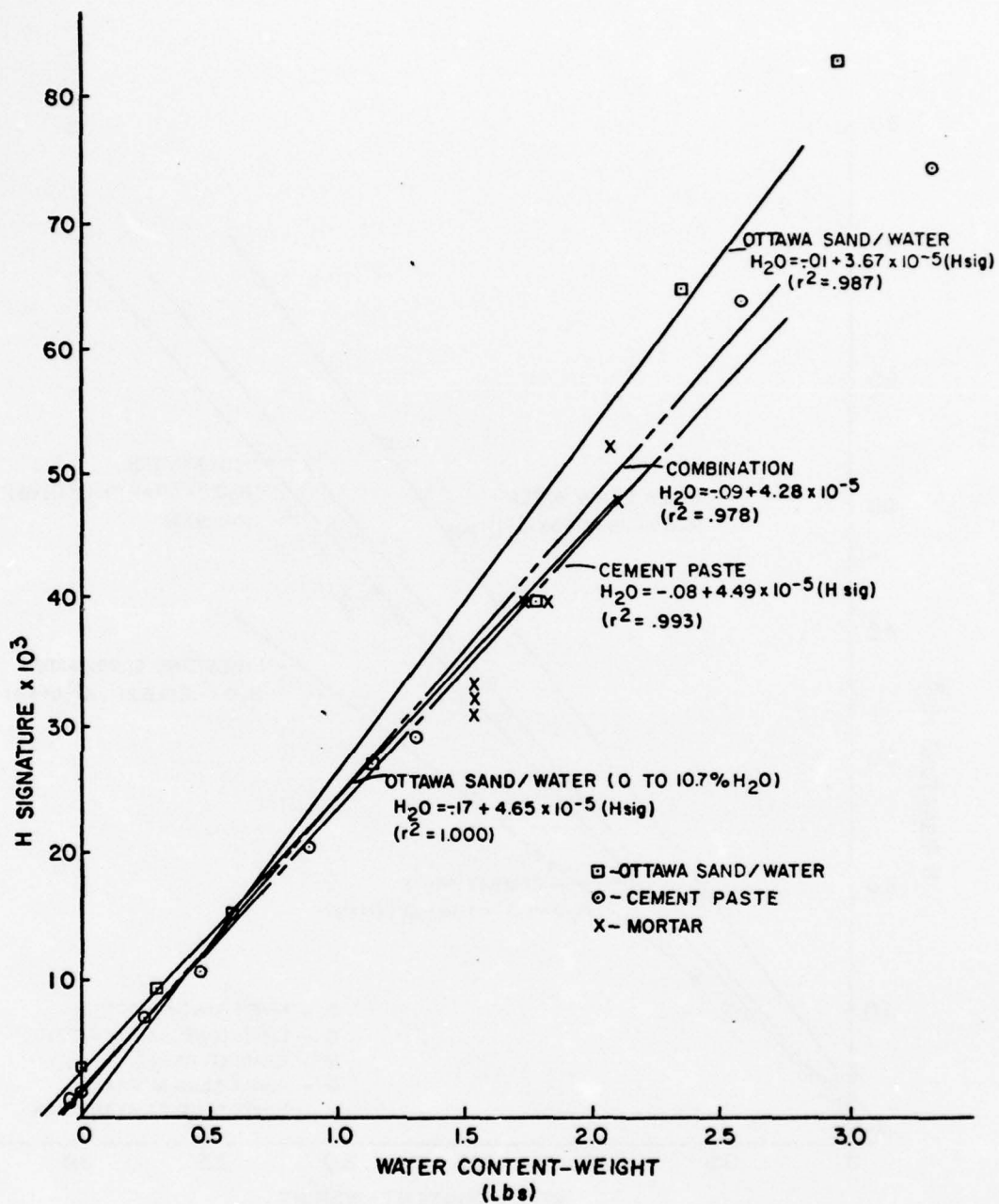


Figure A6. Water content vs. hydrogen signature four-channel hydrogen peak--water content in weight.

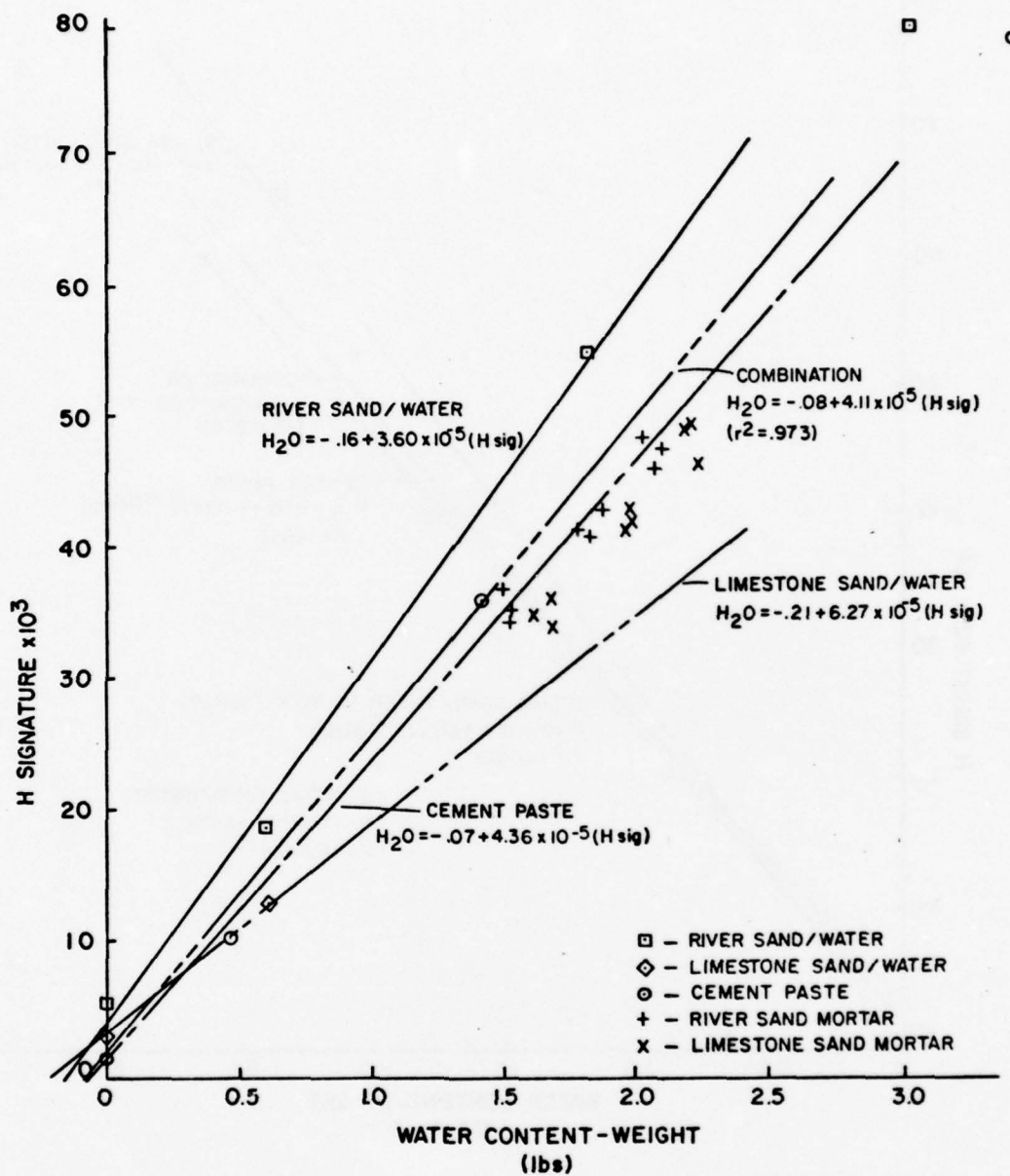


Figure A7. Water content vs. hydrogen signature, four-channel hydrogen peak--water content in weight.

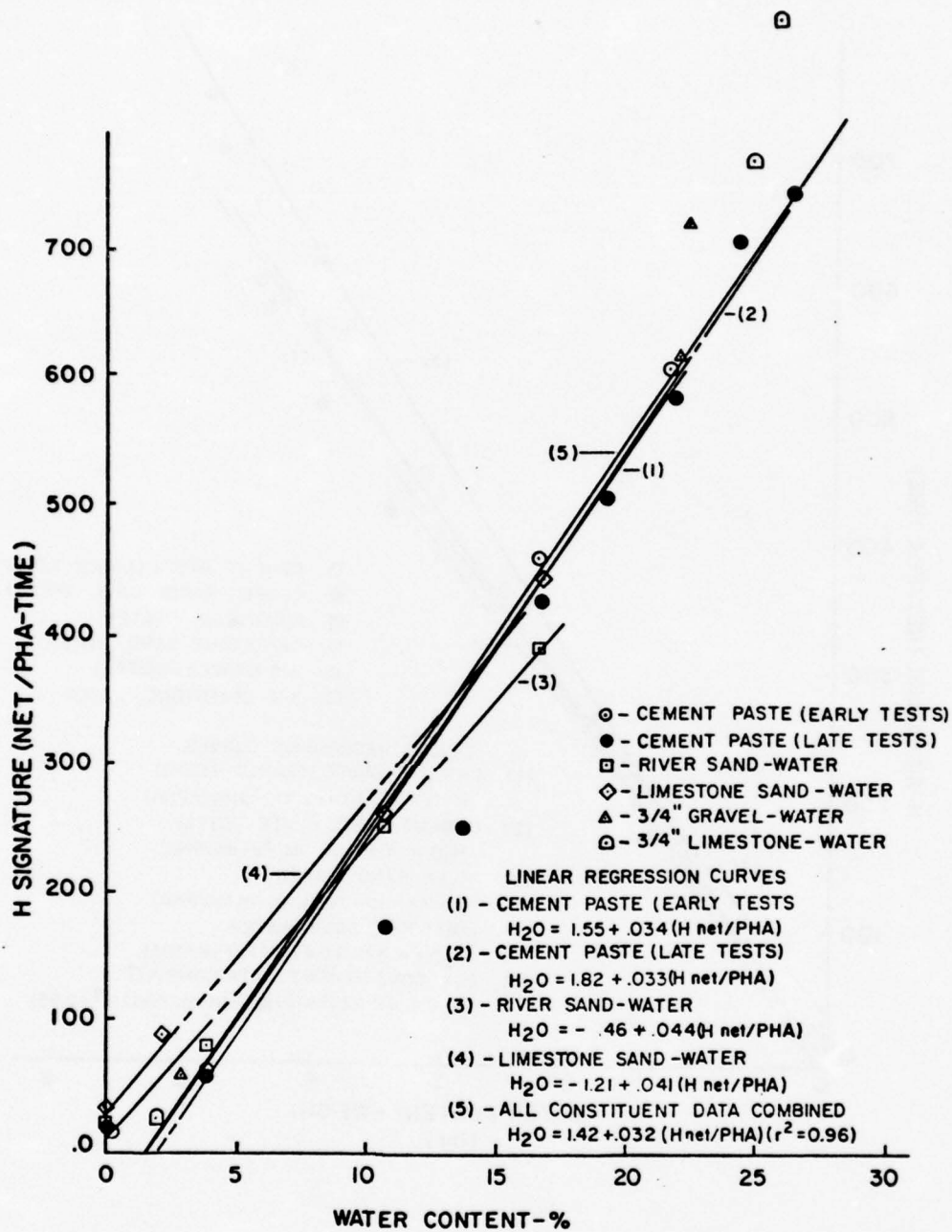


Figure A8. Water content vs. hydrogen signature, concrete constituent data--water content - percent.

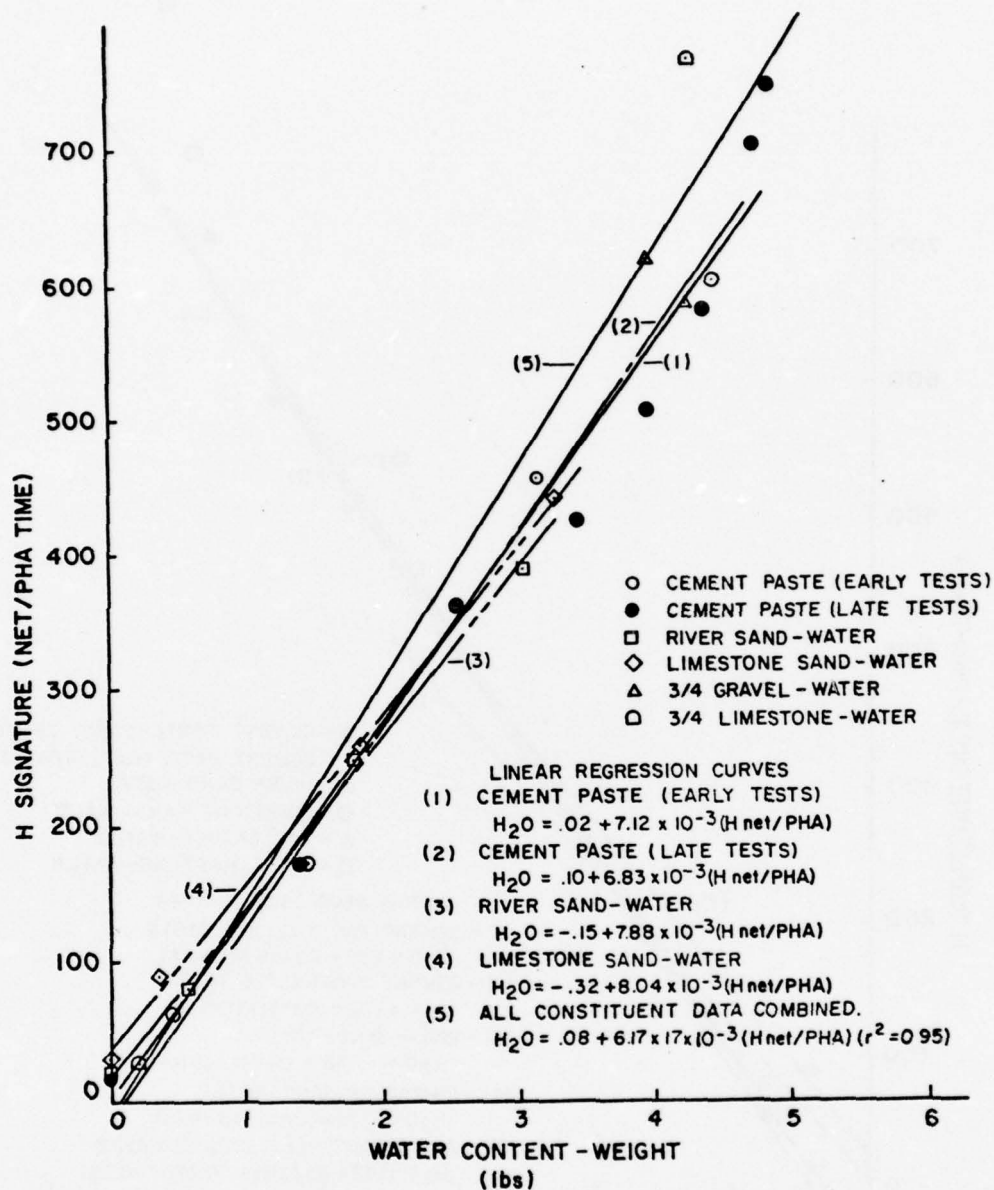


Figure A9. Water content vs. hydrogen signature, concrete constituent data--water content - weight.



the H signature related to water content-weight. The coefficients of determination obtained for the linear regression curves on the combined constituent data in Figures A8 and A9 are nearly equal, .96 and .95, respectively. However, when examining the H signatures and water contents of the concrete tests, it is noted that neither constituent curve is representative of the concrete data (see Figures 24 and 25). The constituent curve for water content percent is significantly more representative of the concrete data than the curve for water content weight. This somewhat counters the previous trend and indicates that the H signature is more closely related to the relative percentage of H present in the sample, rather than the quantity of H actually present.

Since the constituent regression curves did not represent the concrete data, regression curves were computed directly from the concrete data; however, the resulting coefficients of determination were so low (0.20 water content percent and 0.10 water content weight) that little or no confidence should be placed in the resulting curves.

Another set of regression curves was computed from the concrete data by assuming an equal number of data points at the origin. These resulting modified regression curves passed near the origin and through the middle of the data points.

The theoretical accuracy of the H signature was computed by counting statistics. The standard deviations obtained from counting statistics are an upper bound on the accuracy of the neutron/gamma analysis system and are a measure of the count reproducibility for a series of identical tests under identical conditions. Counting statistics are based strictly on the random nature of all nuclear events. In standard deviation units ( $\sigma$ ) the counting error is  $\sigma = \sqrt{n}$ , where  $n$  equals the total count taken. In the presence of background activity, the equation becomes

$$\sigma = \sqrt{n_p + n_b}$$

where  $n_p$  equals the peak count and  $n_b$  equals the background count. The coefficient of variation (C.V.) was also computed.

$$C.V. = \frac{100\sigma}{(n_p - n_b)}$$

and  $n_p - n_b$  is the net peak or signature. The range of counting errors (C.V.) obtained from the H signatures on the concrete data varied from 0.88 to 1.12 percent for net counts that ranged from 43,000 to 57,000.

The actual or operational accuracy of the H signature was determined by conducting a series of repetitive tests on three sets of

aggregate-filled polyester samples. The aggregate fillers consisted of river sand and 3/4-in. (1.90-cm) limestone. Means, standard deviations, and coefficient of variations were computed from the repetitive tests on each of the three sets of polyester samples. Counting errors were also computed for the polyester tests. The counting and repetitive errors in two of the three data sets were in good agreement, and in the third set, the repetitive error was only 3.5 times the counting error (Table A1).

In summary, it can be seen that the H signature is linearly related to the water content of the sample; however, the study could not conclusively determine if the H signature/water content relationship was related to the relative percentage of H (water) present in the sample or to the quantity of H present. The sensitivity of the H signature to other elements in the test sample is minimal if a four-channel net peak is used. For all materials used in this study except the polyester samples, the H signature was almost exclusively related to the sample's water content. The theoretical error in the H signature for concrete type materials was approximately 1 percent, and the operational error based on repetitive tests on a given stable sample varied from less than 1 to more than 3 percent.

#### *Calcium Signature*

The previous paper on the neutron/gamma system indicated that the Ca signature intensities for individual constituents cannot be directly used to estimate peak intensities for mortars.<sup>18</sup> Relative to Ca content, the Ca signatures were much higher for mortars than for the individual constituents. It was suggested that this was due to the presence of H (as water) in the mortar mixes. Hydrogen is an excellent neutron moderator, and increasing hydrogen content increases the tendency for neutron moderation. Thus, when incident neutrons are non-thermal, nuclear reactions that require or favor thermalized neutrons, such as  $^{48}\text{Ca}(n, \gamma)^{49}\text{Ca}$ , become more intense in an increasing H environment.

The polymethyl methacrylate moderator in the TNC system significantly reduces the average kinetic energy of the neutrons from the  $^{252}\text{Cf}$  source, but it can be assumed that many of the neutrons are not completely thermalized when they enter the test sample. Thus, the Ca signature should be sensitive to the presence of water.

The net Ca peaks from the cement paste data (Table 5) are plotted relative to cement content percent (Figure A10). The figure clearly

<sup>18</sup> P. A. Howdysshell, "Preliminary Evaluation of the Neutron/Gamma Technique to Determine the Water and Cement Content of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (CERL, May 1975).

Table A1  
Polyester/River Sand/Limestone - Repetitive Tests

Sample No.	H	Si	C	Al-Si	Ca
1	47443	152429	34786	140418	7643
	47023	147463	34064	140485	7723
	45826	159303	35614	139208	7533
	47360	155507	36426	138823	7208
	46033	162103	35916	133718	7288
Avg =	46737	155361	35361	138530	7479
S.X. =	757	5744	938	2787	223
C.V. =	1.62%	3.70%	2.65	2.01	2.98
2	40881	153189	35107	137931	7724
	41639	147576	35267	137897	7791
	38855	153204	36309	133029	7542
Avg =	40458	151323	35561	136286	7686
S.X. =	1439	3245	653	2820	129
C.V. =	3.56%	2.14	1.84	2.07	1.68
3	40239	153735	34242	127021	7542
	40496	157163	37334	125522	7296
	39968	157543	34857	124085	7381
Avg =	40234	156147	35478	125543	7406
S.X. =	264	2097	1636	1468	125
C.V. =	0.66%	1.34%	4.61	1.17	1.69
Counting Statistics					
S.X. =	469	1567	752	430	114
C.V. =	1.02%	0.98%	2.11%	0.31%	1.51%

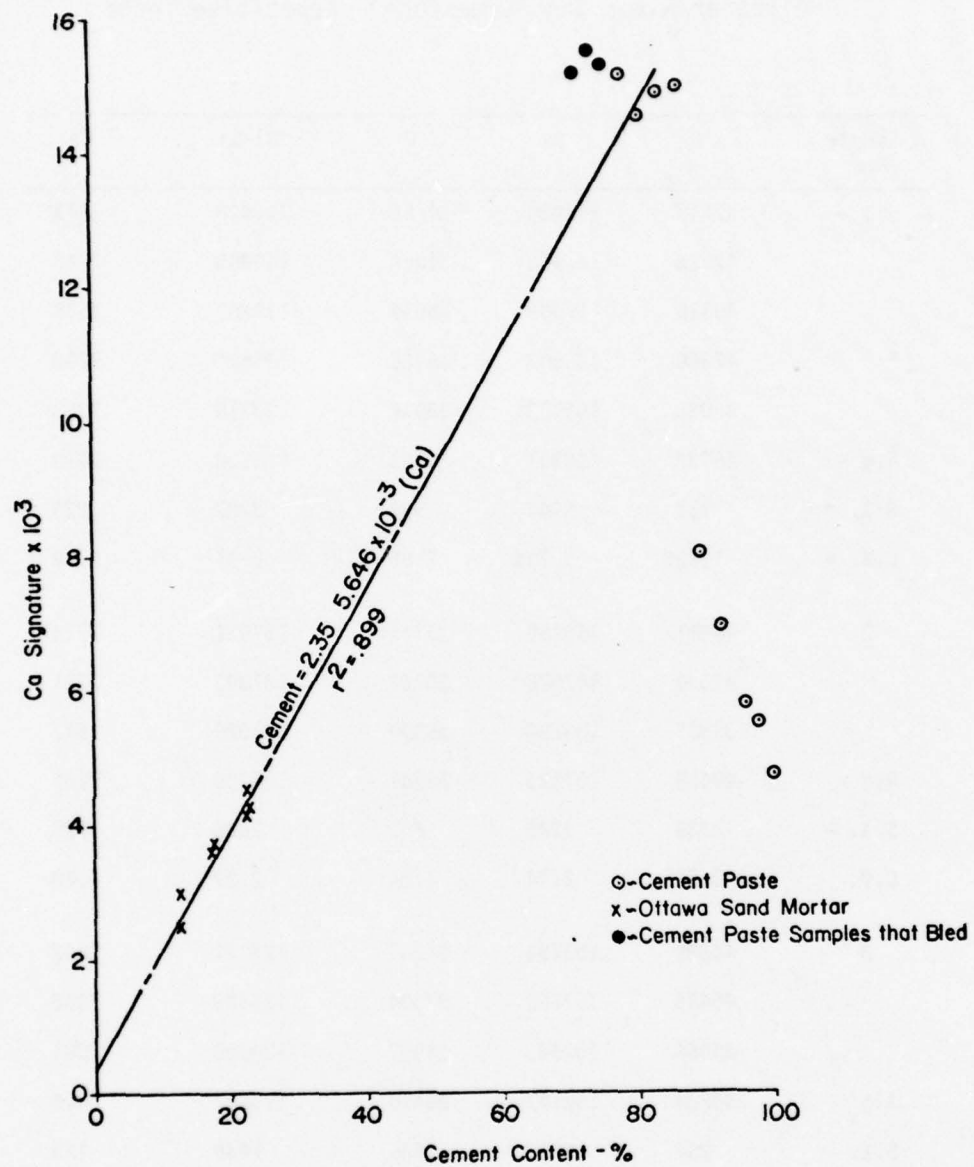


Figure A10. Mortar test series, Ca signature vs. cement content-percent, cement paste and ottawa sand mortars.



Table A1  
Polyester/River Sand/Limestone - Repetitive Tests

Sample No.	H	Si	C	Al-Si	Ca
1	47443	152429	34786	140418	7643
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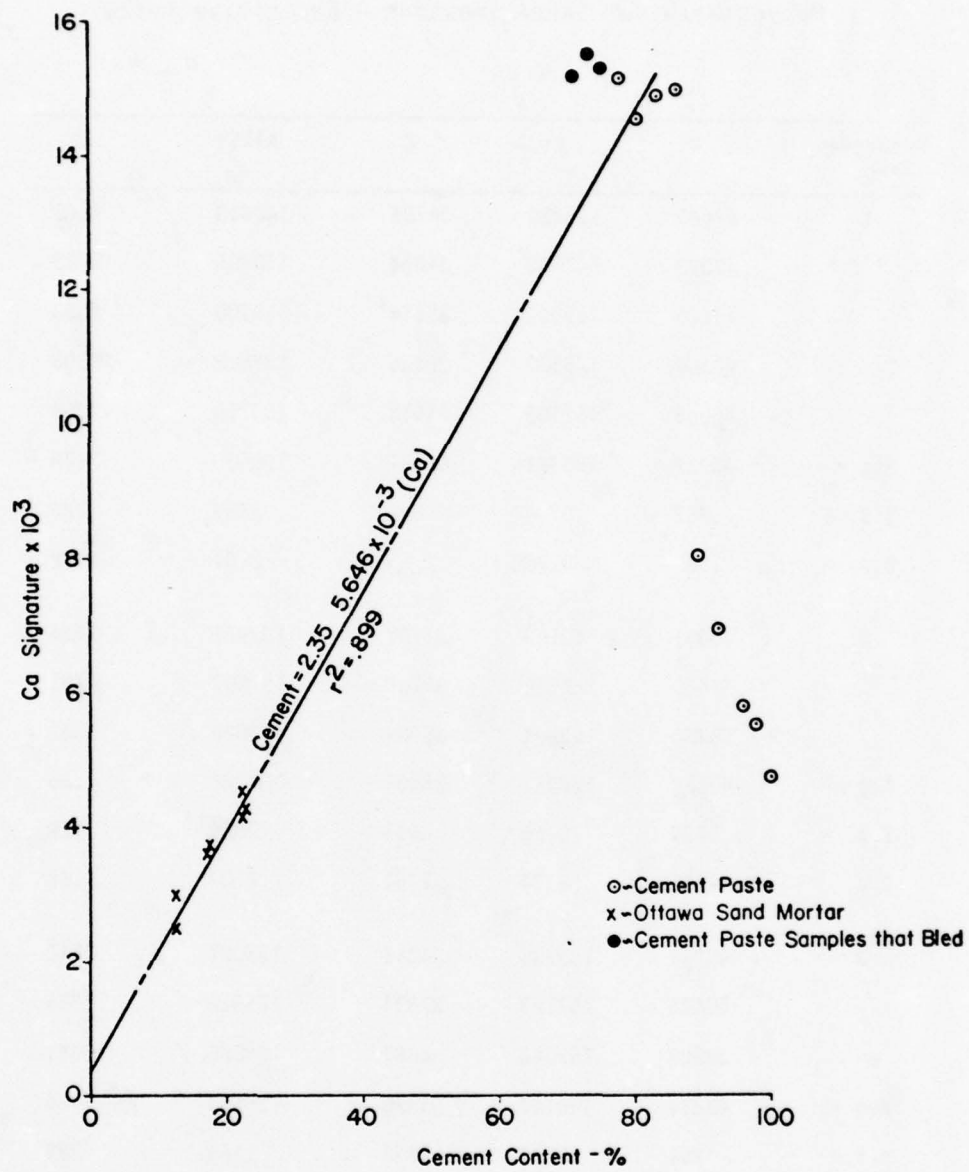


Figure A10. Mortar test series, Ca signature vs. cement content-percent, cement paste and ottawa sand mortars.

illustrates the significant influence that water has on the Ca signature. The cement paste samples having a water content of 13.8 percent or greater have Ca signatures at least three times greater than the Ca signature of the dry cement sample. Noting that increasing the water content of the paste significantly alters the weight and bulk density of the paste samples, the Ca peaks from the cement paste data are also plotted relative to the weight of cement in the paste sample (Figure A11). These results clearly illustrate that the higher Ca signatures are not caused solely by the increased cement quantities.

The net Ca peaks from the ottawa sand mortar data (Table 8) are also plotted in Figures A10 and A11. The ottawa sand constituent tests (Table 6) indicate that ottawa sand does not contain any measurable amount of Ca. Thus, the mortar Ca peaks should be entirely associated with the Ca in the cement. Linear regression curves are developed for the ottawa sand mortar data relating the Ca signature to cement content. The regression analysis is developed both for cement content percent (Figure A10) and weight (Figure A11). In both cases (Figures A10 and A11), the projections of the regression lines intersect the cement paste data within or near the paste samples containing 13.8 percent water or more. The regression lines also pass very close to the origin, thus indicating a negligible Ca signature for zero percent cement.

Net Ca peaks for the cement paste (Table 5) and the ottawa sand mortars (Table 8) are divided by cement content percent (Ca multiplier percent) and plotted relative to water content percent (Figure A12) and water content-weight (Figure A13). Similarly, the Ca peaks are divided by cement content-weight (Ca multiplier-weight) and plotted relative to water content percent (Figure A14) and water content-weight (Figure A15). The four figures illustrate that the Ca multipliers are sensitive to the presence of water; however, when the mortar samples are compared to the paste samples, the sensitivity does not appear to be linear for any of the four combinations plotted.

The same Ca multipliers are also plotted relative to water/cement ratios. Figure A16 is the plot of the Ca multipliers percent versus water/cement ratios, and Figure A17 is the plot of the Ca multipliers weight versus water/cement ratios. Again, the figures illustrate the sensitivity of the Ca multipliers to water or water/cement ratio; however, if the mortar samples are included in the comparison, no simple linear relationship is apparent.

If both the paste samples that exhibit bleeding and those that contain less than a .16 water/cement ratio are dropped from the comparison, the water contents or water/cement ratios do not drastically influence the Ca multipliers. The high coefficients of determinations obtained from the regression curves for the mortar data (Figures A10 and A11) also illustrate that within the range of water contents used

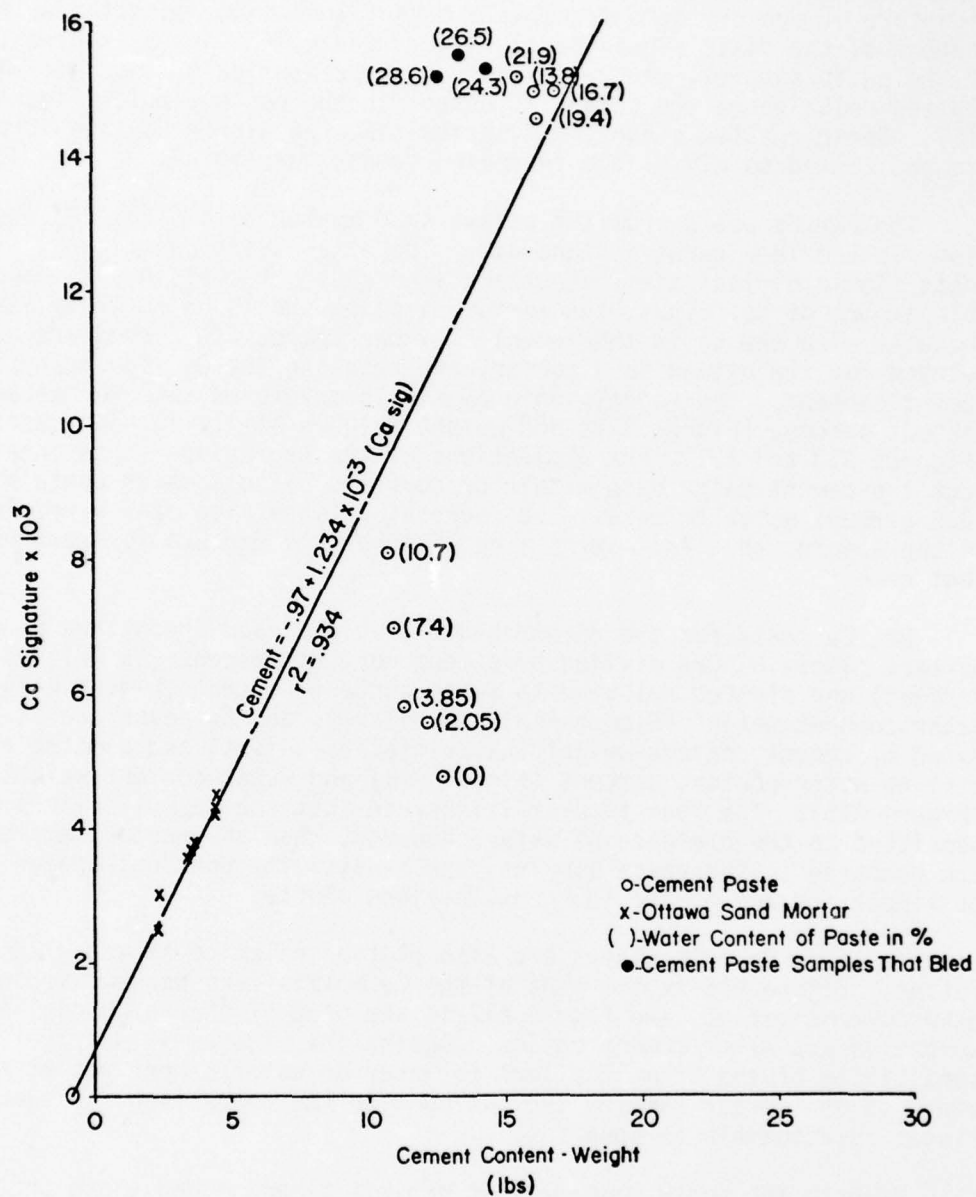


Figure A11. Mortar test series, Ca signature vs. cement content (lbs), cement paste and ottawa sand mortars.



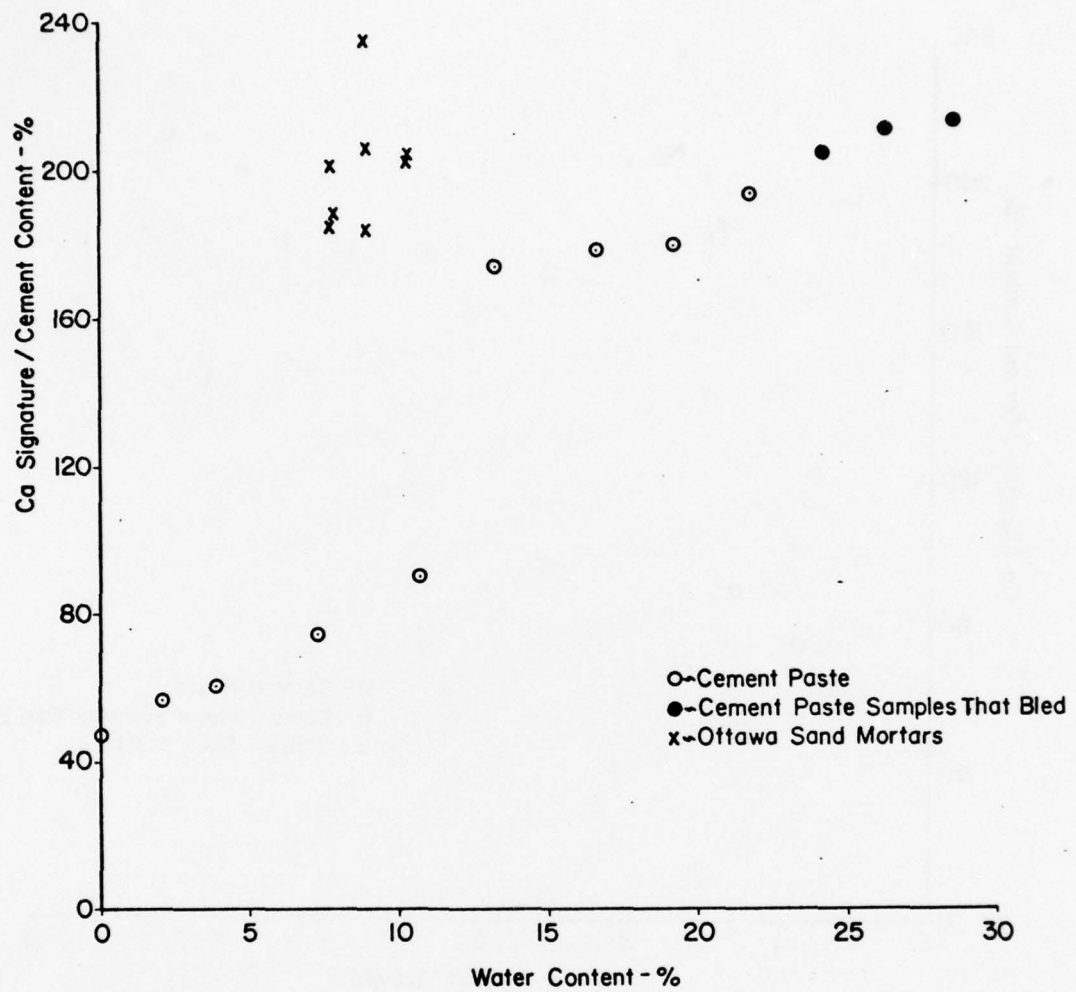


Figure A12. Mortar test series, Ca multiplier - percent weight vs. water content percent, cement paste and ottawa sand mortars.

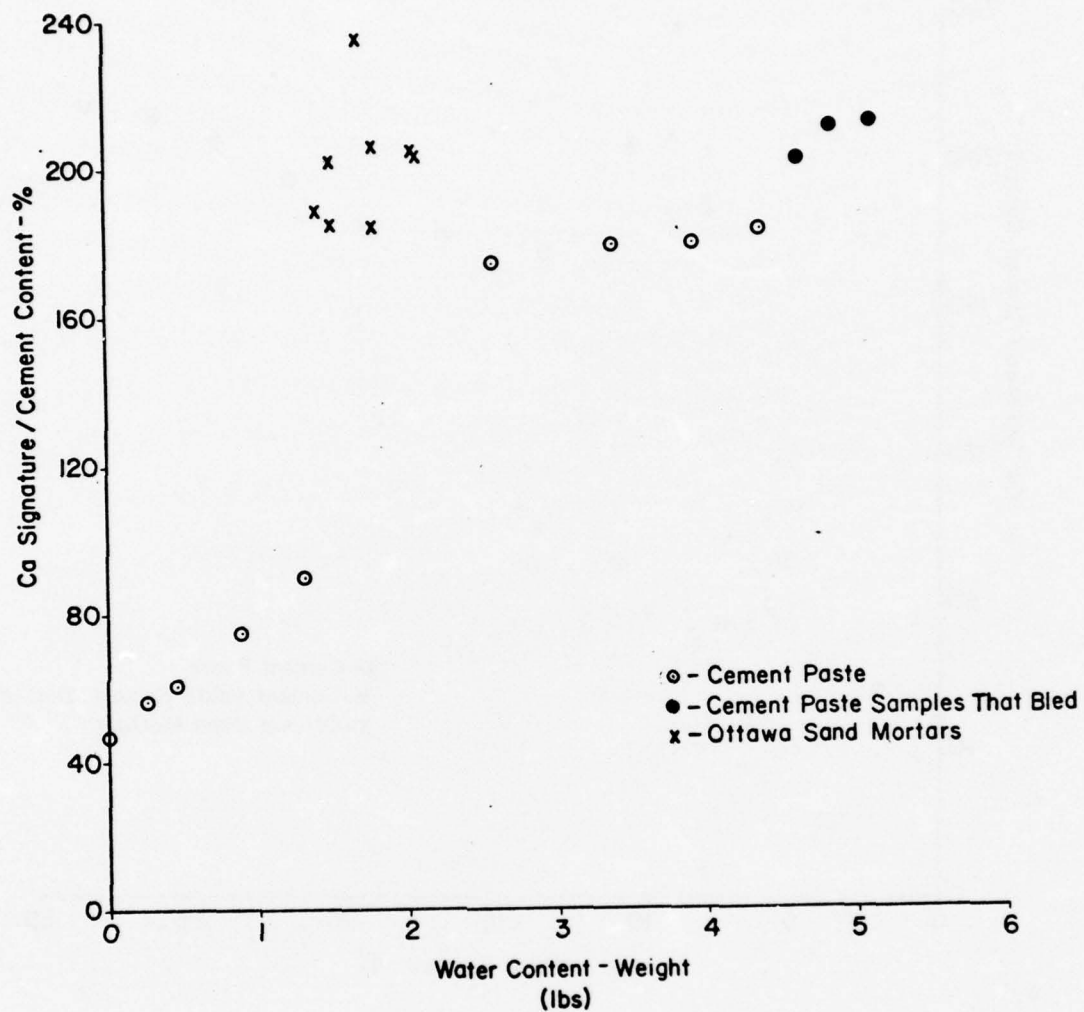


Figure A13. Mortar test series, Ca multiplier - percent weight vs. water content weight, cement paste and ottawa sand mortars.

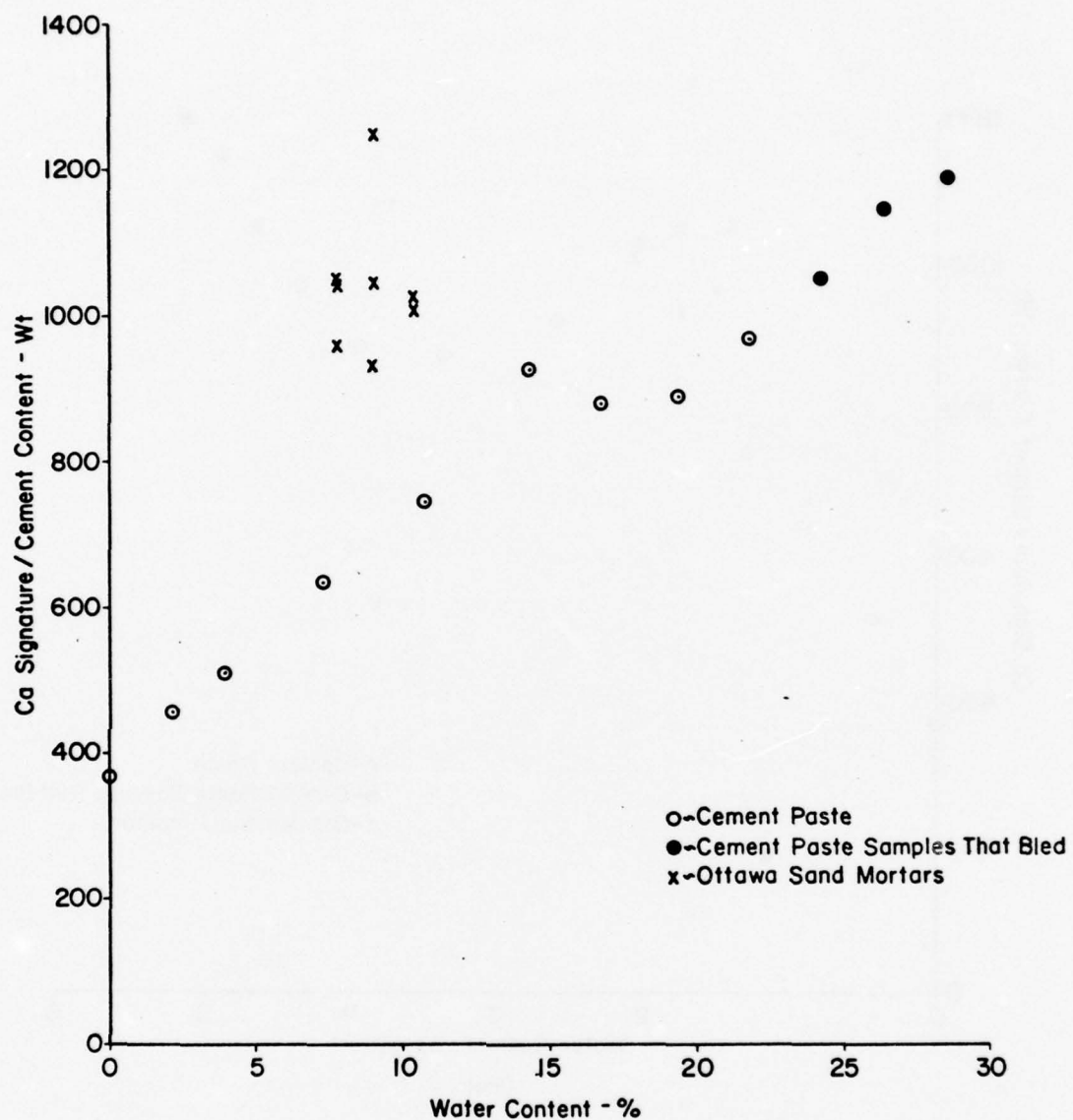


Figure A14. Mortar test series, Ca multiplier - weight vs. water content percent, cement paste and ottawa sand mortars.

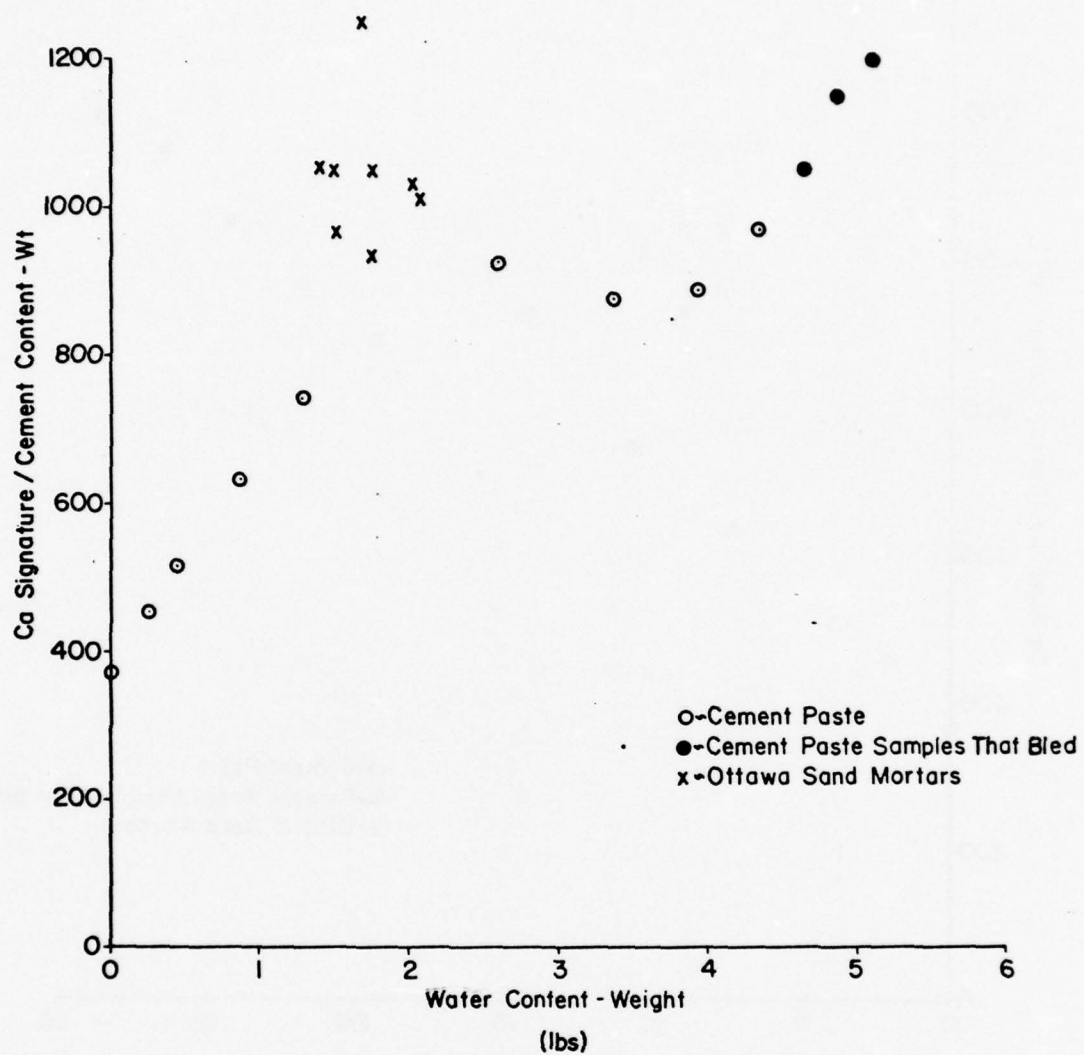


Figure A15. Mortar test series, Ca multiplier - weight vs. water content weight, cement paste and ottawa sand mortars.



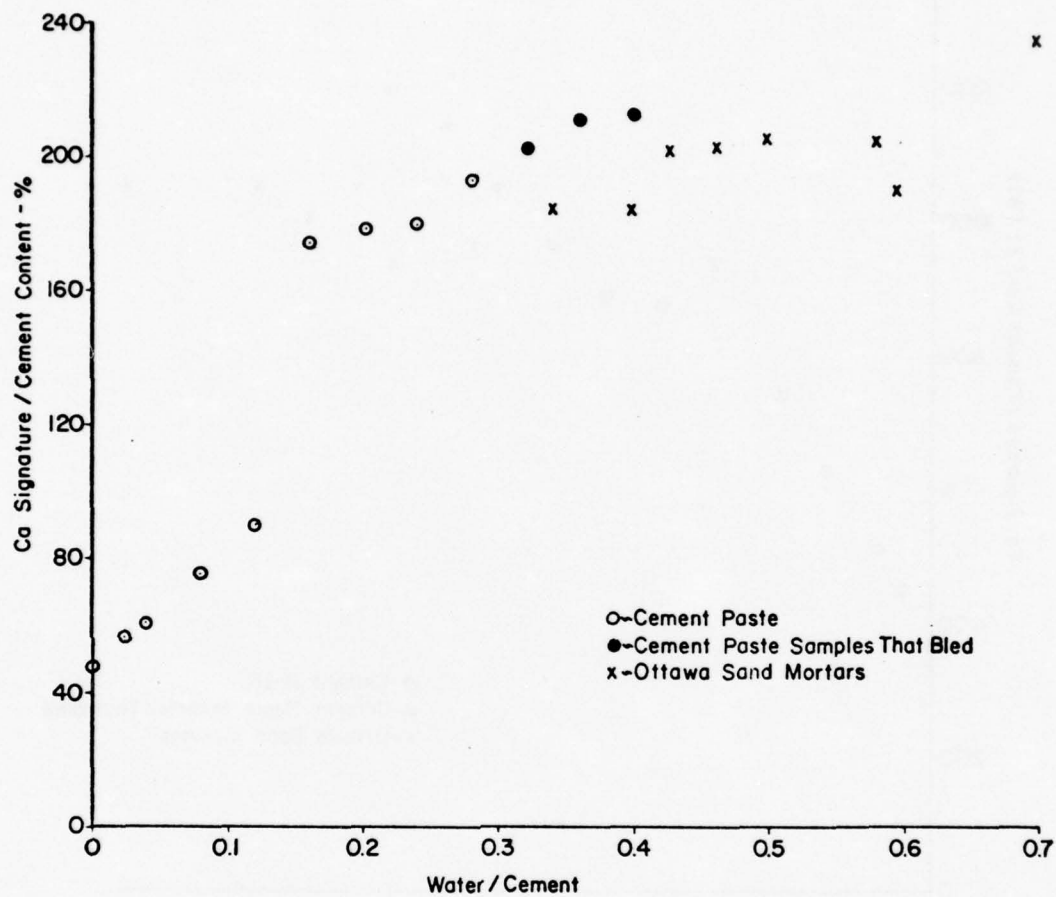


Figure A16. Mortar test series, Ca multiplier - percent vs. water/cement ratio cement paste and ottawa sand mortar.

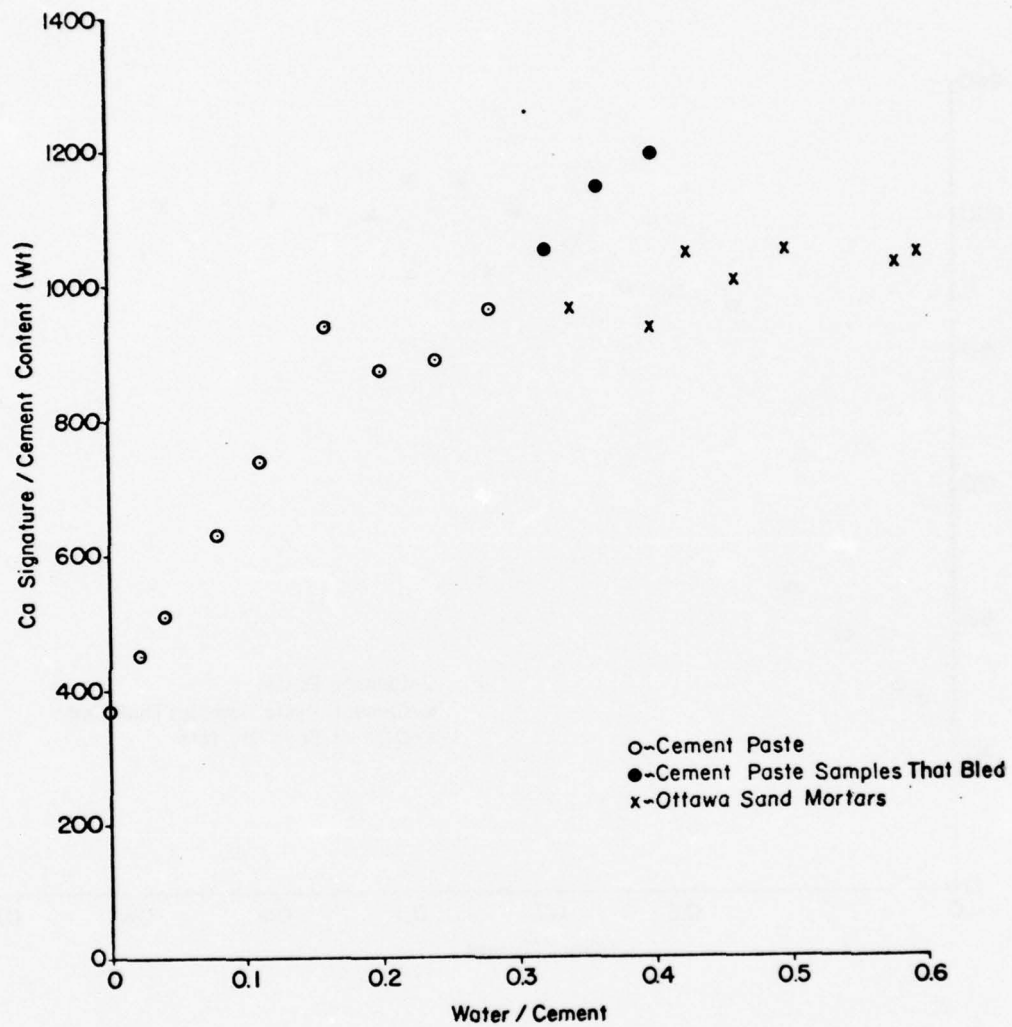


Figure A17. Mortar test series, Ca multiplier - weight vs. water/cement ratio cement paste and ottawa sand mortars.

(10.4 and 7.9 percent), the Ca signatures and their associated multipliers are only slightly sensitive to variations in water content; however, there is a drastic difference in Ca sensitivity between anhydrous samples and samples containing water. (See Table A2 for a comparison of the Ca multipliers for dry constituents versus constituent water samples.)

Figure A18 is the plot of the Ca signature versus constituent content percent for the river sand mortars and the related constituents, and Figure A19 is the Ca signature versus constituent contents percent for the limestone sand mortars and related constituents. Both the river and limestone sands contain some Ca (Table 7), so a simple comparison of regression lines through the mortar data is meaningless. The Ca multipliers for both the cement and sand can be determined from the mortar tests if: (1) there are two or more sand-cement ratios for each water content; and (2) it is assumed that the Ca signature from a sand-cement mixture simply follows the law of mixtures (cement content multiplied by its Ca multiplier plus the sand content multiplied by its Ca multiplier equals the Ca signature of the mortar). The two Ca multipliers can be determined by plotting the Ca signatures of the mortars relative to both cement and sand content (Figures A18 and A19). Linear regressions are developed for the data that relate to a constant sand plus cement content. In these cases, the regression lines are developed for the 7.9, 9.1, and 10.4 percent water contents. The intersection of the Ca signature-cement regression line with the zero constituent content axis is the Ca signature if the sand-cement mixture is entirely sand. For example, the 9.1 percent water content cement regression line intersects the zero constituent content axis at 2000 counts. The Ca multiplier for sand would therefore be:

$$2000/90.9 = 22.0 \text{ counts/percent sand}$$

where 90.9 = constant sand plus cement content (also 100 minus water content of mortar tests).

Likewise, the intersection of the Ca signature-sand regression line with the zero constituent content axis is the Ca signature if the sand-cement mixture is entirely cement.

Table A3 lists the cement, river, and limestone sand Ca multipliers that were computed from the mortar tests. The multipliers fluctuated significantly with varying water contents, but no distinct trends were observed in the fluctuations. The average of the cement multipliers obtained from the river sand mortars was approximately equal to that obtained from the limestone-sand mortars.

Table A2  
Comparison of Ca Multipliers  
Dry Constituents Vs. Constituent/Water Vs. Mortars

	Dry	Water Contents	Mortar
Cement Paste		13.8-21.9% H <sub>2</sub> O	O.S.M.*
Ca Sig/Cement-%	47.1	181.55	201.175
% of Dry Sample	100.0	385.0	427.0
River Sand		10.7-16.7% H <sub>2</sub> O	
Ca Sig/Sand-%	18.03	28.55	18.7
% of Dry Sample	100.0	158.0	104.0
Limestone Sand		10.7% H <sub>2</sub> O	
Ca Sig/Sand-%	64.02	155.5	146.5
	100.0	243.0	229.0
Cement Paste		16.7-21.9% H <sub>2</sub> O	R.S.M.* L.S.M.*
Ca Sig/Cement-%wt	56.2	201.2	205.7 199.5
	100.0	358.0	366.0 355.0

\* R.S.M. = River Sand Mortar  
L.S.M. = Limestone Sand Mortar  
O.S.M. = Ottawa Sand Mortar

Table A3  
Ca Multipliers Obtained From River Sand  
and Limestone Sand Mortars

Ca Multiplier	% H <sub>2</sub> O			
Water	7.9	9.1	10.4	Aug
RSM* River Sand	5.18	21.4	29.5	18.7 counts/%
RSM Cement	241.1	200.2	175.7	205.7 counts/%
LSM* Limestone Sand	183.0	129.8	126.6	146.5 counts/%
LSM Cement	40.6	265.6	292.4	199.5 counts/%

\* RSM = River Sand Mortar  
LSM = Limestone Sand Mortar



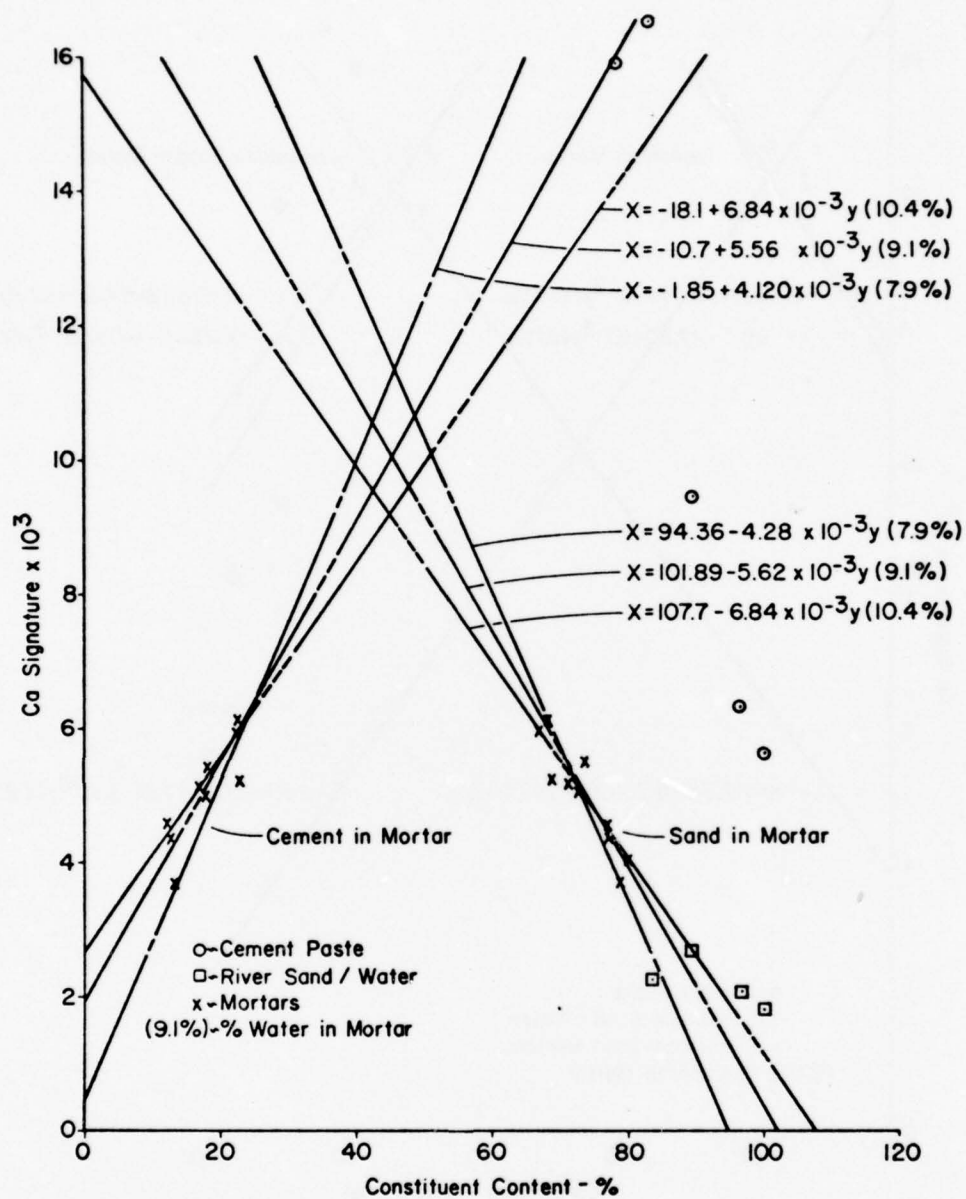


Figure A18. Mortar test series, Ca signature vs. constituent contents, cement paste, river sand/water and river sand mortars.

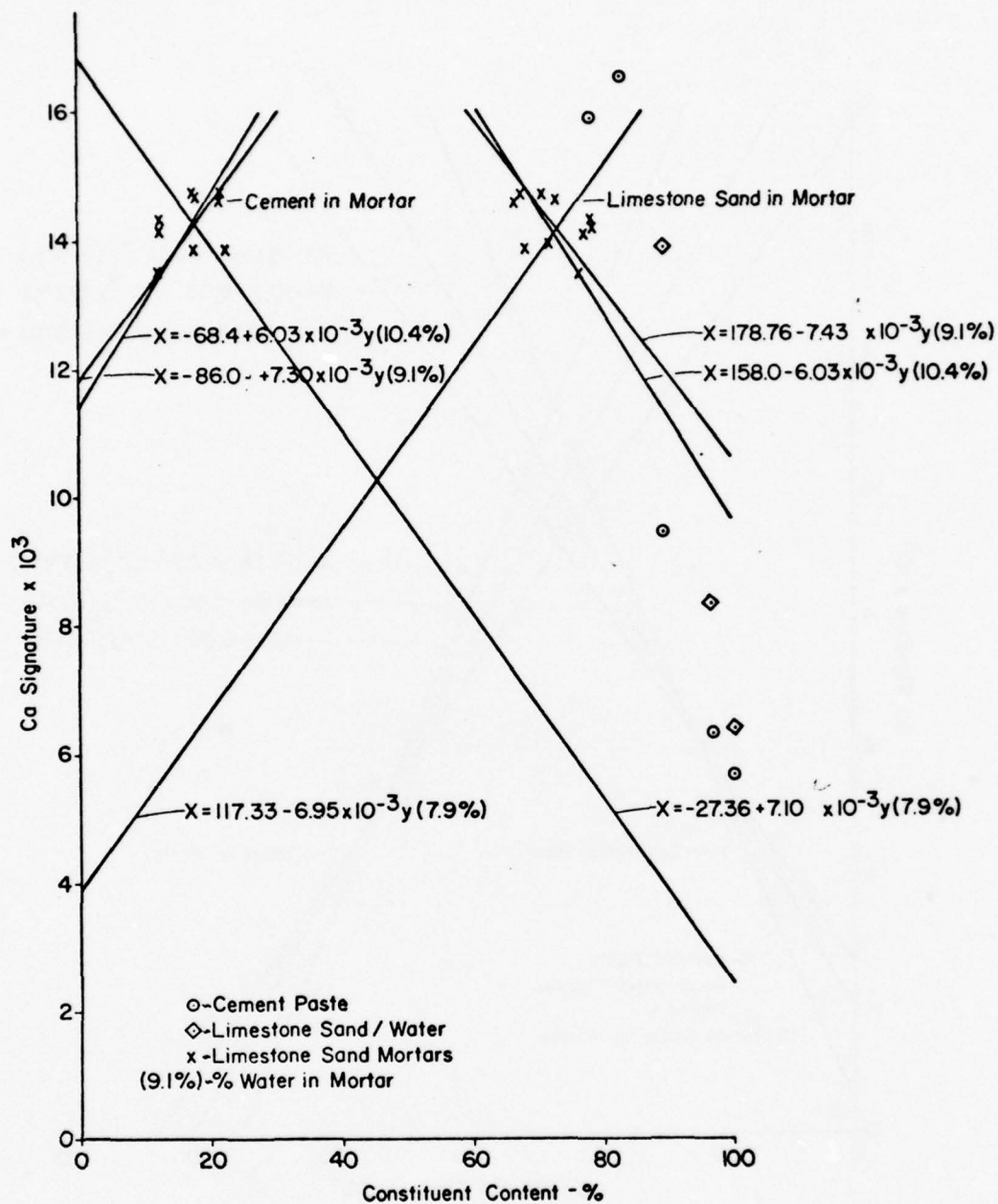


Figure A19. Mortar test series, Ca signature vs. constituent contents, cement paste, limestone sand/water and limestone sand mortars.

Comparing the Ca multipliers obtained from the individual constituent-water tests to those obtained for the mortar tests (Table A2) indicates reasonable agreement between the cement multipliers for the three mortars and the cement multipliers for the cement paste tests containing 13.8 percent or more water. Likewise, the limestone-sand Ca multipliers obtained from the mortar and constituent-water tests agree reasonably well. The river sand Ca multipliers exhibited far less agreement, but this may be attributed to the relative small size of the river sand Ca multiplier.

Another factor that may have caused some of the data scatter observed in the mortar and associate constituent tests was that for the vast majority of cases the first run of the day normally produced smaller Ca signatures than did subsequent runs (Tables 5 through 10). This phenomenon can be observed throughout the mortar test series. It occurred even though the electrical circuits had been on for more than 1 hour, and at least one dummy sample had been irradiated before testing began. This problem was resolved by leaving the power and all electronics on 24 hours a day during the concrete and associated constituent tests.

In addition, since the PHA times of the mortar and associated constituent tests fluctuated slightly, all computation on the concrete test series used signature rate intensities (net signature count divided by PHA time) instead of simple signature intensities.

The concrete and associated constituent tests were analyzed to determine constituent multipliers based on rate intensities. Three techniques were used to generate the multipliers. The first was referring to the constituent water data and dividing the resulting Ca-signature intensities in this data by the constituent contents of the samples. Constituent contents were computed as both percent and sample weight. The mortar tests proved that the Ca signature is sensitive to the presence of H, but that its sensitivity becomes negligible for the range of H (water) contents common to concrete. Thus, in computing an average Ca multiplier from the constituent-water data, only water contents of more than 13 percent were used.

The second method was applying multiple linear regression analysis directly to the concrete data; however, since the coarse aggregate content of the concrete samples did not change significantly, the resulting best fit multipliers varied unrealistically from very large positive numbers to negative values. A second series of linear regressions was run where upper and lower bounds were placed on the resulting multipliers. Normally the bounds were chosen as  $\pm 10$  to 20 percent of constituent test-derived multipliers. In some cases the bounds were increased further until the sum of the predicted Ca signature equaled that of the actual Ca signatures.

The third method was applying the linear regression technique used in the mortar data analysis. (This method assumed that the coarse aggregate and water contents of the mixes did not vary.) The result was two equations describing the three unknown multipliers. To obtain an exact solution, the ratio of the coarse to fine aggregate multipliers obtained from the constituent-water data was introduced as a third condition.

Table A4 contains resulting Ca multipliers obtained from the three methods of computation. In the majority of cases, the multipliers obtained from the constituent-water data were greater than those obtained from either technique for analyzing the concrete data. Furthermore, the difference was even greater when the multipliers were computed on a weight basis instead of a percent basis.

Counting statistics were used to compute the theoretical accuracy of the Ca signature. The range of counting errors (C.V.) obtained from the Ca signature on the concrete test data varied from 1.12 percent for a net 12,775 counts to 2.30 percent for a net 4840 counts. The actual operational accuracy of the Ca signature was computed from the repetitive tests on the aggregate-filled polyester samples. The C.V. obtained from the multiple tests on the three sets of polyester samples were 2.98, 1.68, and 1.69 percent. The C.V. obtained from the counting statistics for the same samples averaged 1.51 percent (Table A1). Thus, the operational accuracy of the system is very close to its upper-bound theoretical accuracy of 1 to 2 percent.

In summary, the data indicate that the Ca signature (and signature ratio intensity) is linearly related to the sample's Ca content. The sensitivity of the Ca signature to elements other than H (water) that are common to concrete appears to be negligible. The signature's sensitivity to H is significant when anhydrous samples are compared to samples containing water; however, when compared to the range of water contents common to mortars and concrete, the signature's sensitivity to H is negligible. There does not appear to be any single or simple relationship between the Ca signatures for individual dry constituents (cement, fine aggregates, and coarse aggregates) and their associated signatures when combined with water. The theoretical error in the Ca signature for concrete-type materials varied from 1.0 to 2.3 percent, and the occupational error based on repetitive tests on a given stable sample varied from 1.5 to 3.0 percent.

#### *Aluminum-Silicon Signature*

The paper on the neutron/gamma system<sup>19</sup> indicated that the Al-Si signature intensities obtained from individual constituents were

<sup>19</sup> P. A. Howdyshell, "Preliminary Evaluation of the Neutron/Gamma Technique to Determine the Water and Cement Content of Fresh Concrete," *Rapid Testing of Fresh Concrete*, Conference Proceedings M-128/ADA009702 (CERL, May 1975).



Table A4  
Concrete Test Series  
Constituent Multipliers

		Ca		Al-Si		C		Si	
		Counts Per %	Counts Per lbs	Counts Per %	Counts Per lbs	Counts Per %	Counts Per lbs	Counts Per %	Counts Per lbs
Cement	Constituent - Water	0.77	3.92	12.18	61.92	0.0	0.0	2.60	17.06
	x Concrete Mix 1-5	0.87	3.23	13.38	64.03	0.0	0.0	2.56	11.60
	x Concrete Mix 6-10	0.68	2.77	10.98	43.82	0.0	0.0	2.34	10.43
	x Concrete Mix 11-15	0.70	2.71	9.78	42.00	0.0	0.0	2.60	11.34
	x Concrete Mix 16-20	0.65	2.30	11.61	45.52	0.0	0.0	2.30	12.30
	+ Concrete Mix 1-5	1.22	--	25.22	--	0.0	--	2.17	--
	+ Concrete Mix 6-10	0.72	--	11.42	--	.21	--	2.42	--
	+ Concrete Mix 11-15	0.71	--	5.68	--	.32	--	2.84	--
	+ Concrete Mix 16-20	0.71	--	12.29	--	0.0	--	2.07	--
River Sand	Constituent - Water	0.06	0.71	10.40	59.56	0.04	0.28	6.45	41.05
	x Concrete Mix 1-5	0.05	.52	9.40	45.00	0.03	0.31	7.05	31.40
	x Concrete Mix 6-10	0.05	.50	9.40	42.00	0.03	0.22	6.07	27.03
	+ Concrete Mix 1-5	0.03	--	8.02	--	0.04	--	6.73	--
	+ Concrete Mix 6-10	0.10	--	9.41	--	0.04	--	5.99	--
Limestone Sand	Constituent - Water	0.50	2.56	11.03	53.18	0.20	1.24	2.28	14.09
	x Concrete Mix 11-15	0.40	1.50	10.29	43.50	0.18	1.00	2.05	9.21
	x Concrete Mix 16-20	0.40	2.04	9.93	42.58	0.22	1.32	2.51	10.65
	+ Concrete Mix 11-15	0.40	--	11.49	--	0.14	--	2.00	--
	+ Concrete Mix 16-20	0.40	--	9.82	--	0.49	--	2.45	--
3/4 Gravel	Constituent - Water	0.22	1.28	10.68	58.57	0.12	0.78	4.57	29.53
	x Concrete Mix 1-5	0.19	0.97	10.00	44.00	0.14	0.74	4.18	20.00
	x Concrete Mix 16-20	0.21	1.02	9.70	48.03	0.11	0.62	4.78	21.26
	+ Concrete Mix 1-5	0.07	--	8.02	--	0.10	--	4.57	--
	+ Concrete Mix 16-20	0.19	--	9.51	--	0.0	--	4.91	--
3/4 Limestone	Constituent - Water	0.72	4.12	2.83	16.26	0.27	1.90	1.47	10.12
	x Concrete Mix 6-10	0.67	2.90	2.74	14.28	0.25	1.50	1.33	6.09
	x Concrete Mix 11-15	0.59	2.96	2.23	12.05	0.24	1.50	1.33	6.00
	+ Concrete Mix 6-10	0.62	--	2.56	--	0.21	--	1.36	--
	+ Concrete Mix 11-15	0.71	--	2.95	--	0.19	--	1.29	--

NOTE: x-- multiple linear regression analysis  
+-- simple linear regression analysis

lower than those obtained from mortar tests. Again it is assumed that H is a moderator. Thus, when incident neutrons are nonthermal, nuclear reactions that require or favor thermalized neutrons become more intense in an increasing H environment. The Al-Si signature, in which two reactions are involved, is unique. The  $^{27}\text{Al}(n, \gamma)^{28}\text{Al}$  activation reaction favors thermalized neutrons; the  $^{28}\text{Si}(n, p)^{28}\text{Al}$  transmutation reaction requires that the interacting neutrons have energies in excess of 2 MeV. Thus, the sensitivity of the Al-Si signature to water content is expected to be very complex, and to be dependent on both the relative quantities of Al and Si present and the energy of the incident neutrons.

The net Al-Si peaks for the cement paste and ottawa sand data (Tables 5 and 6) have been plotted relative to constituent content percent (Figure A20) and constituent content-weight (Figure A21). Both figures illustrate the influence that water has on the Al-Si signature for cement paste. Conversely, the ottawa sand-water tests show very little, if any, H influence and the Al-Si signature appears to be related to sand content percent. Cement contains 4 percent Al and 10 percent Si. Ottawa sand contains 46 percent Si and only trace levels of Al. It is assumed that the majority of neutrons that pass through the polymethyl methacrylate moderator from the Cf source have energies less than 2 MeV. Thus, as illustrated by the cement paste and ottawa sand-water constituent tests, the moderated neutrons significantly favor the  $^{27}\text{Al}(n, \gamma)^{28}\text{Al}$  reaction over the  $^{28}\text{Si}(n, p)^{28}\text{Al}$  reaction. Also, the  $^{27}\text{Al}(n, \gamma)^{28}\text{Al}$  reaction is sensitive to water content, whereas the  $^{28}\text{Si}(n, p)^{28}\text{Al}$  reaction does not appear to be.

To further illustrate the influence of water content on the Al-Si signatures, Al-Si multipliers (net peaks divided by constituent content) for the constituent-water tests are computed and plotted relative to water content. Figures A22 and A23 are the Al-Si multipliers-weights to water content percent and water content weight, respectively. Figures A24 and A25 are the Al-Si multipliers percent to water content percent and water content weight, respectively. Figures A22 through A25 illustrate the influence of water on the Al-Si signatures for cement and river sand. The Al-Si signature for limestone sand also appears to be sensitive to water, but the data base is insufficient to illustrate the water influence clearly. As indicated previously, the Al-Si multipliers for ottawa sand appear to be insensitive to the presence of water. The figures also illustrate that at water contents greater than 3 lb (1.2 kg) or 12 percent, the Al-Si multipliers for cement and river sand become less sensitive to variations in water content.

The Al-Si multipliers are computed from the three mortar test series in a manner similar to that used to compute Ca multipliers from mortar data. Figure A20 contains the ottawa sand mortar data and regression lines, and Figures A26 and A27 contain the river and

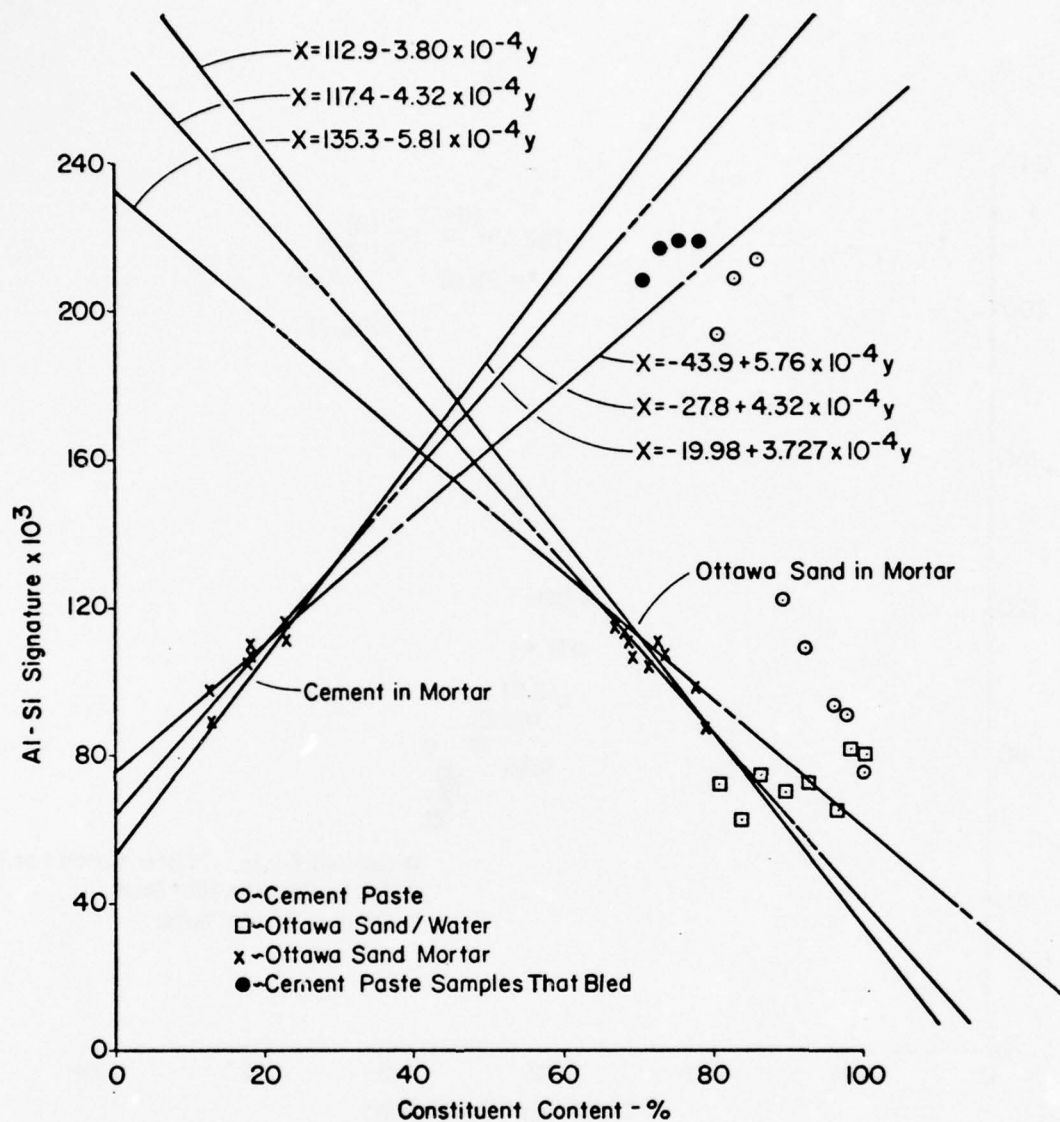


Figure A20. Mortar test series, Al-Si signature vs. constituent content - cement paste, ottawa sand/water, ottawa sand mortars.

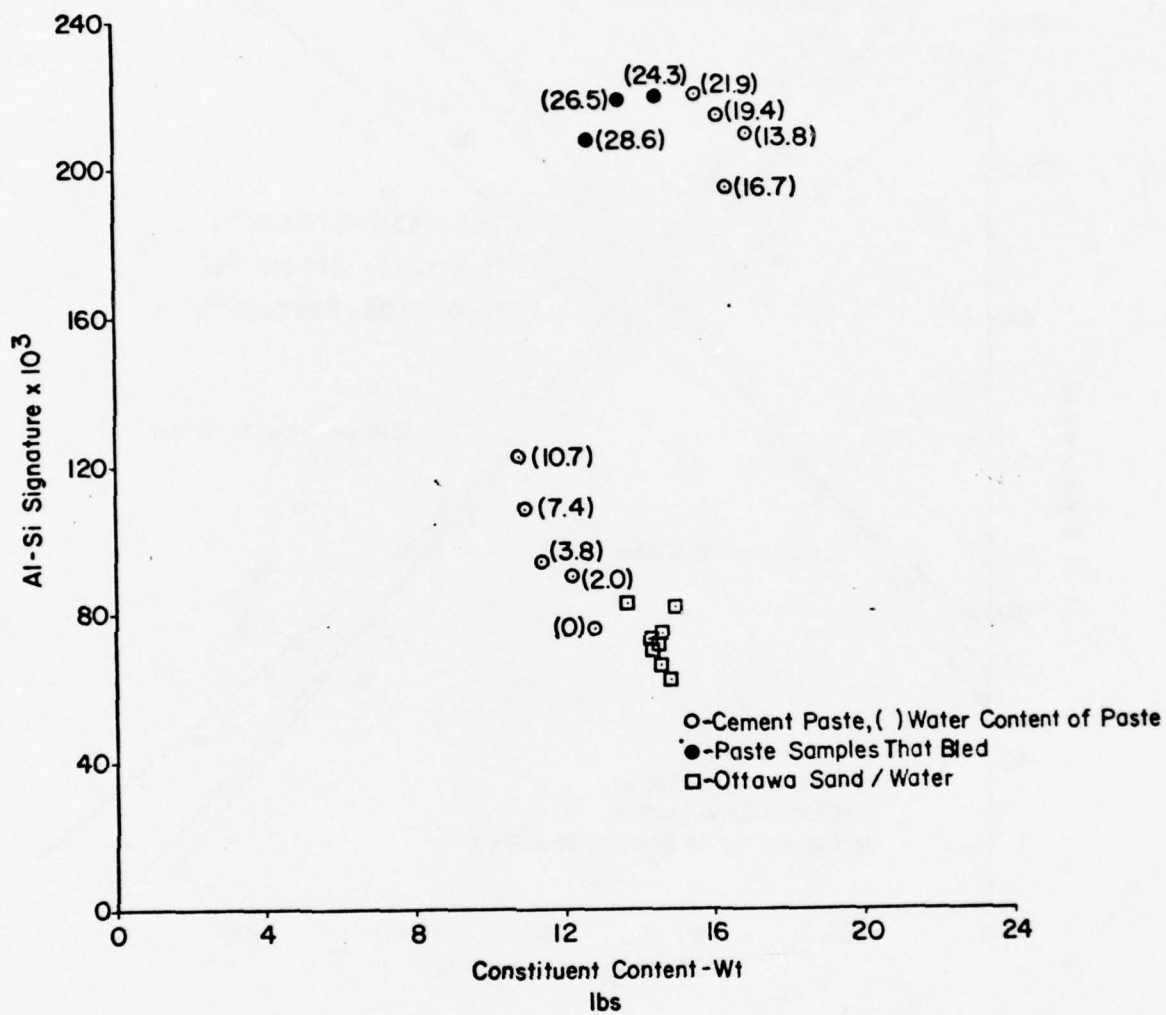


Figure A21. Mortar test series, Al-Si signature vs. constituent content - weight cement paste and ottawa sand/water.



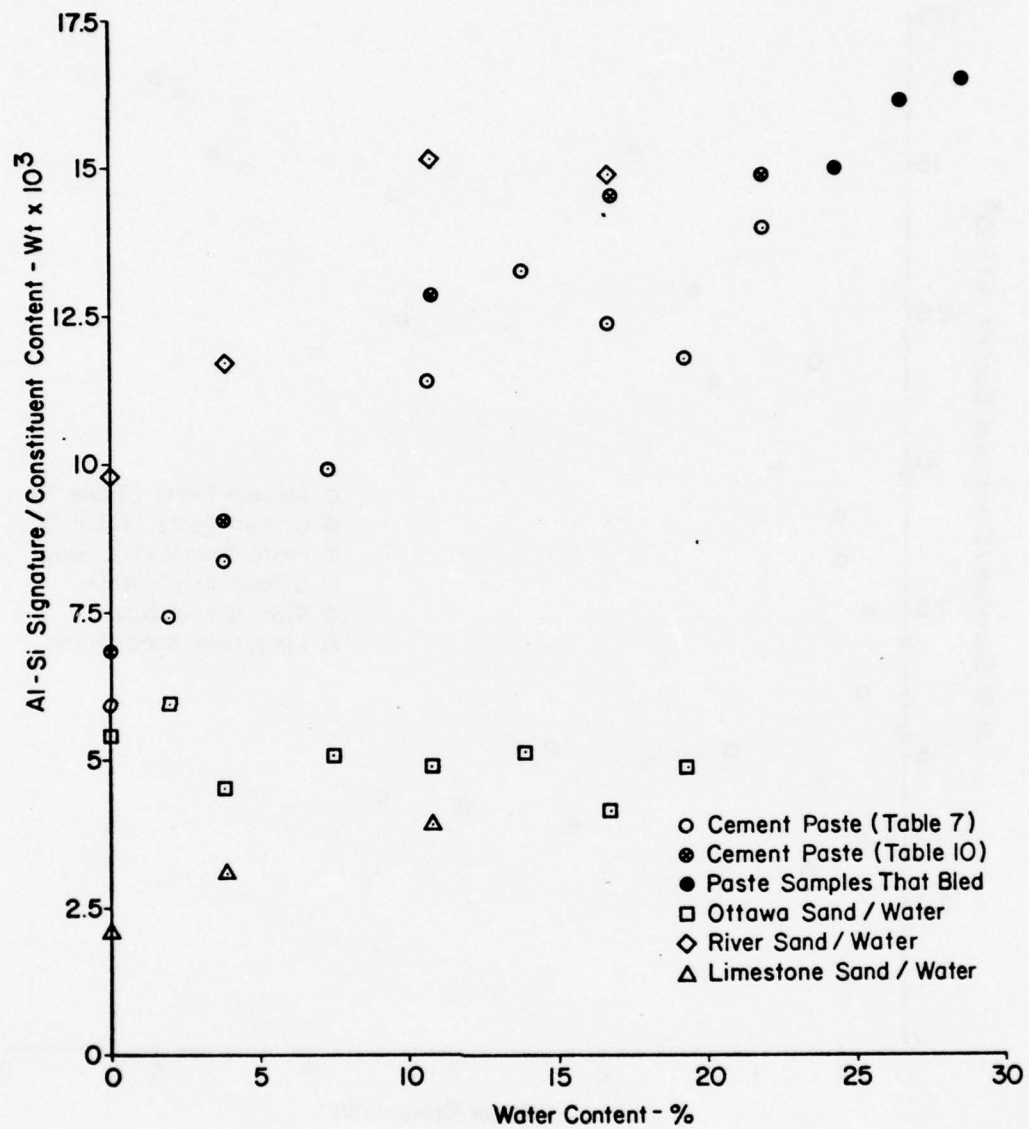


Figure A22. Mortar test series, Al-Si multiplier - weight vs. water content - percent constituent/water data.

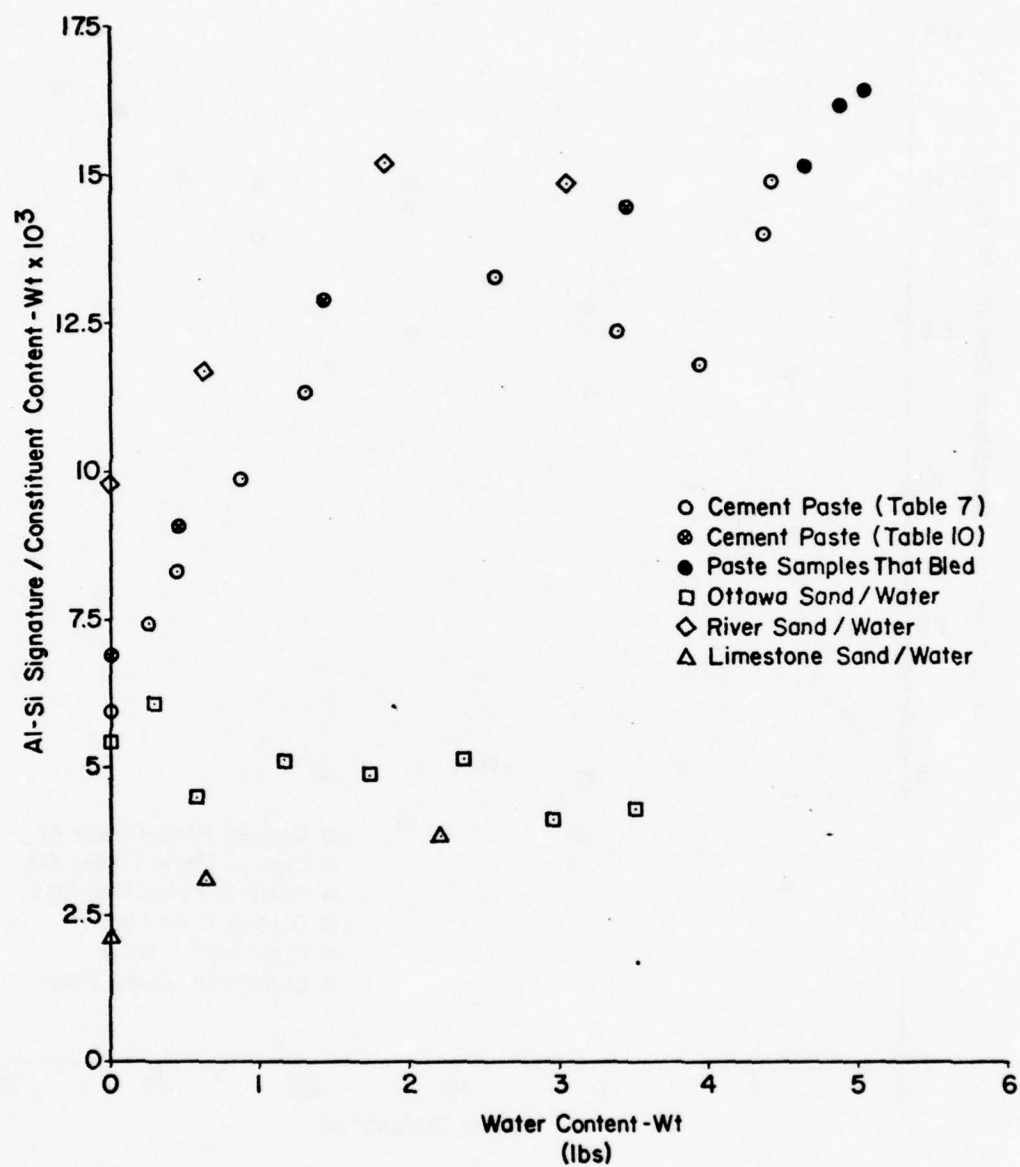


Figure A23. Mortar test series, Al-Si multipliers - weight vs. water content - weight constituent/water data.

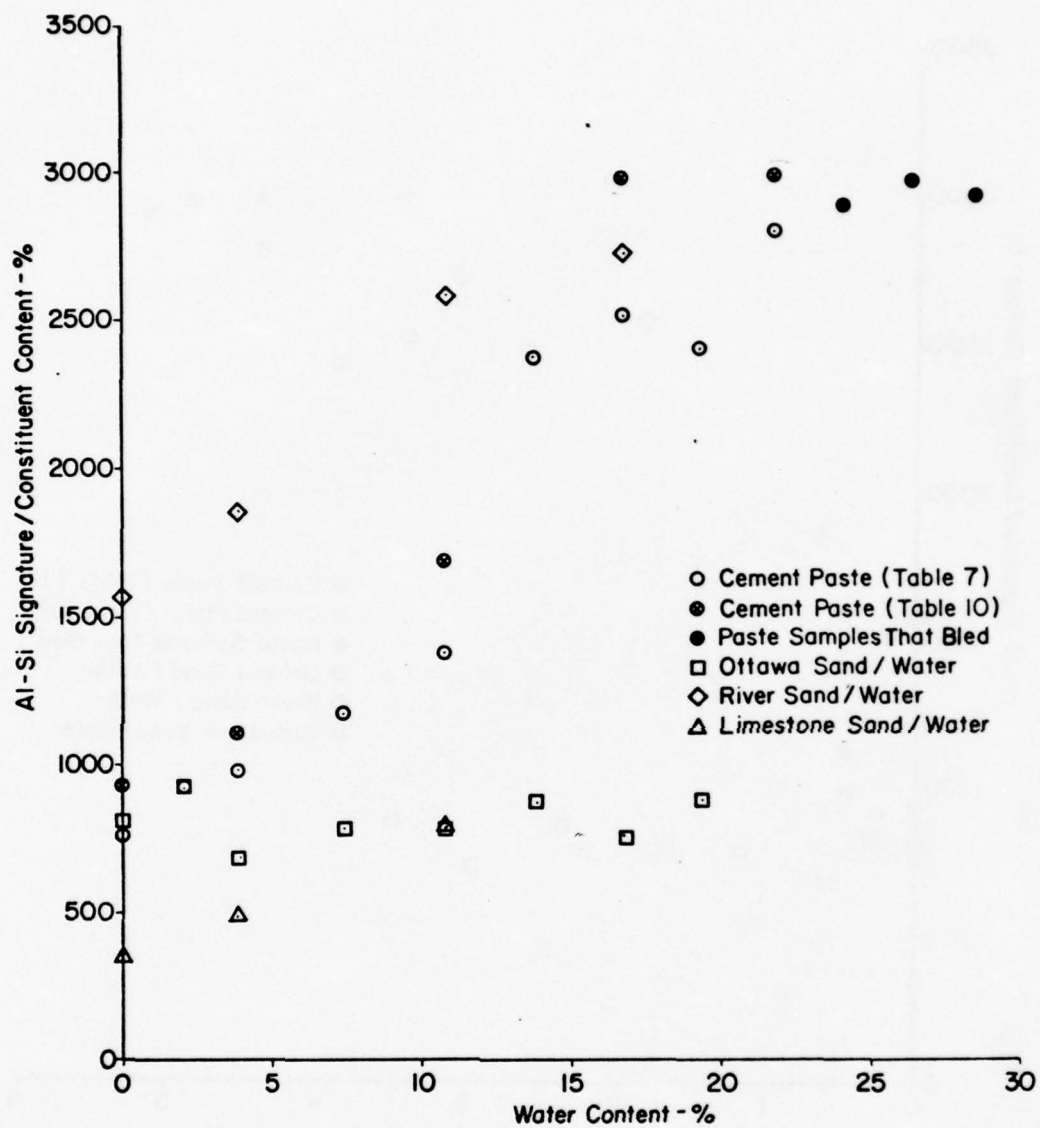


Figure A24. Mortar test series, Al-Si multiplier - percent vs. water content - percent constituent/water data.

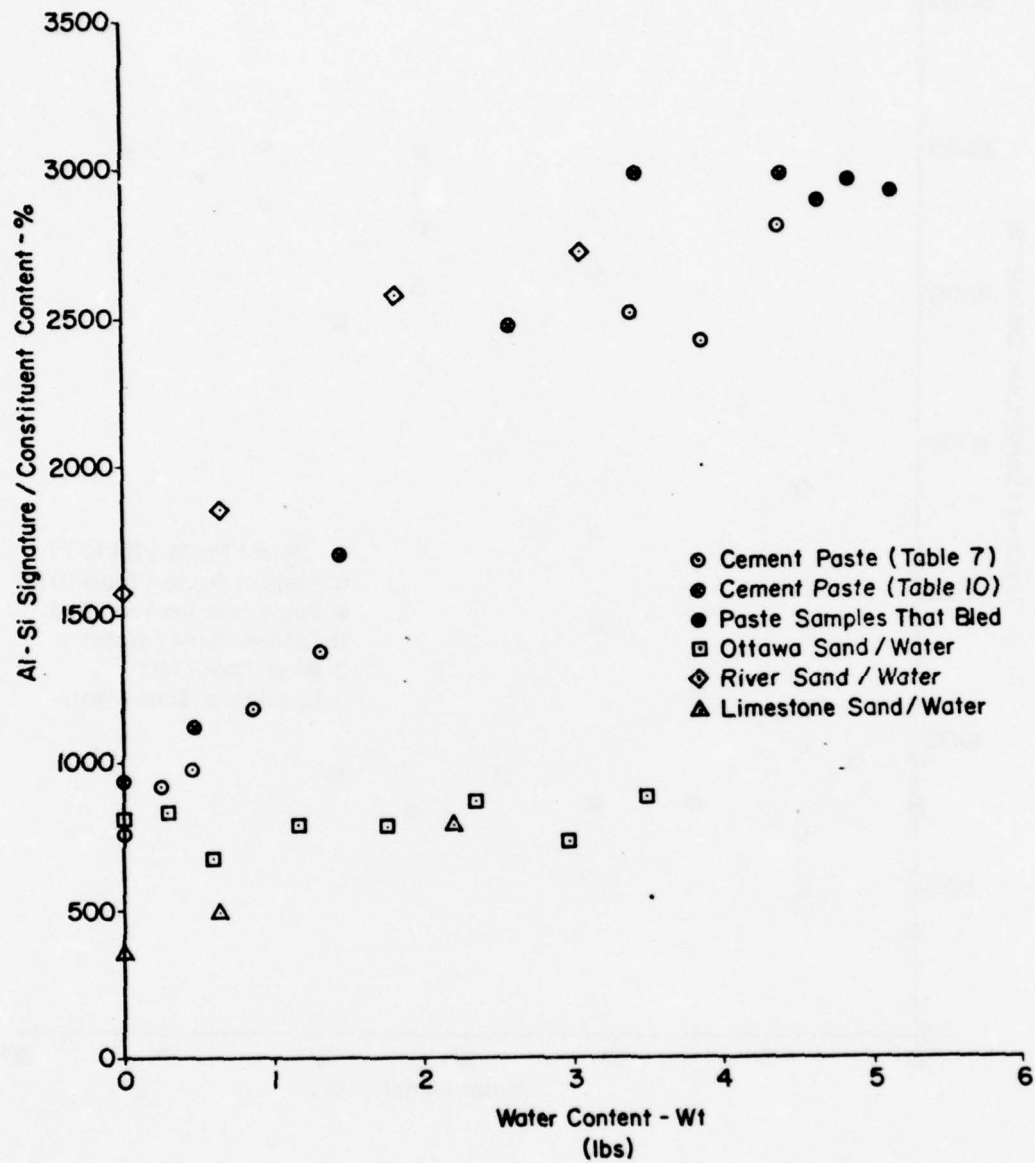


Figure A25. Mortar test series, Al-Si multiplier - percent vs. water content - weight constituent/water data.



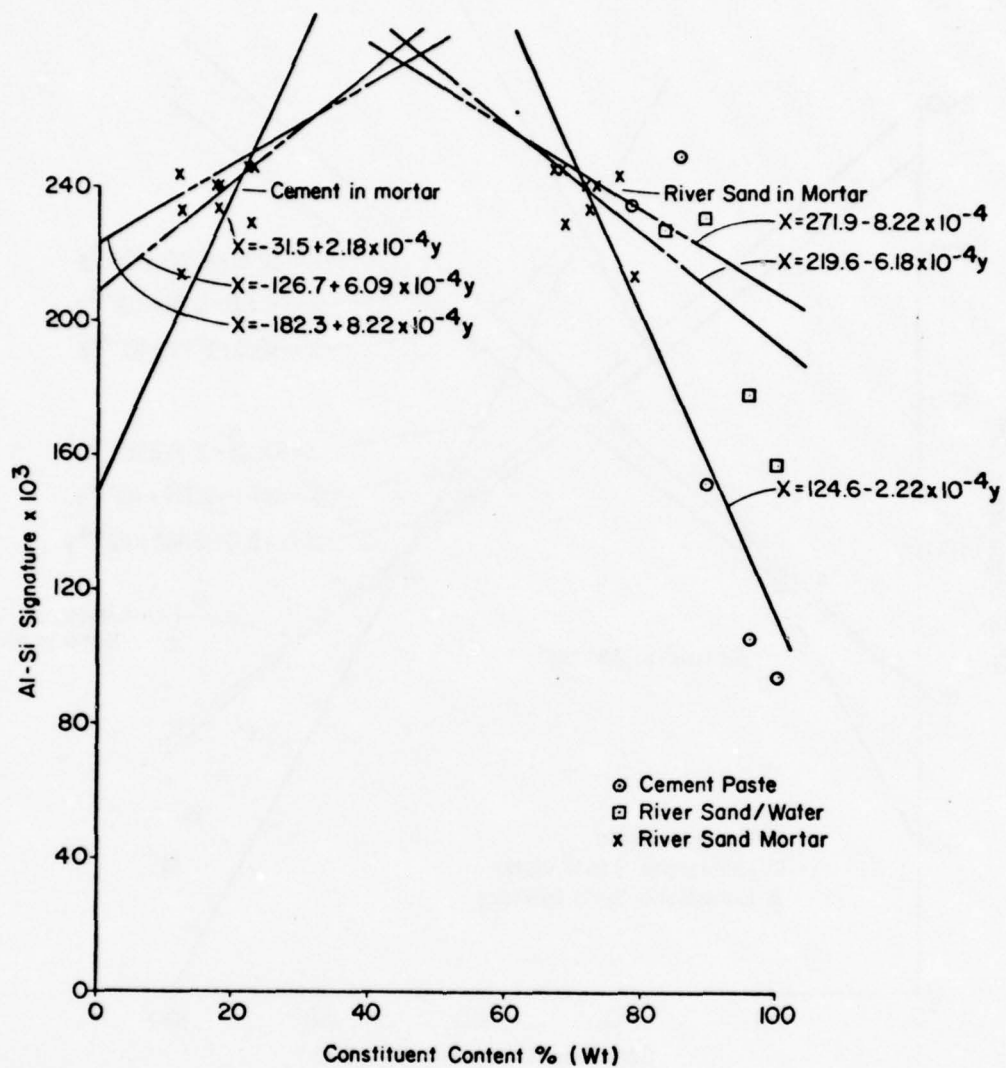


Figure A26. Mortar test series, Al-Si signature vs. constituent content - percent weight cement paste, river sand water, and river sand mortar.

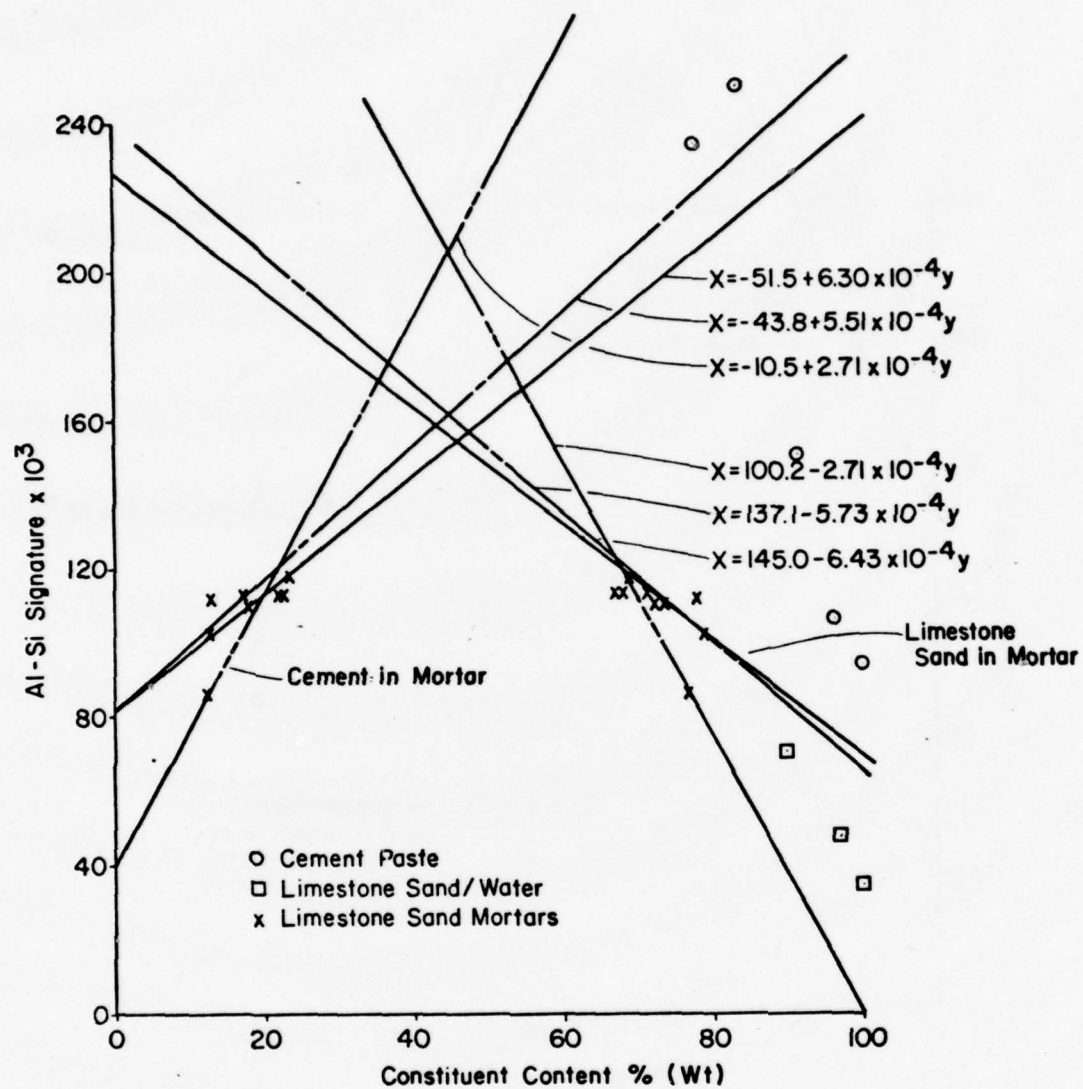


Figure A27. Mortar test series, Al-Si signature vs. constituent content - percent weight cement paste, limestone sand water, and limestone sand mortars.

limestone sand mortar data and regression lines, respectively. Table A5 lists the cement multipliers and the Ottawa, river, and limestone sands Al-Si multipliers that were computed for the three levels of water content in the mortar tests. The multipliers fluctuate significantly with varying water contents, but no distinct trends have been observed. The average computed cement multipliers obtained from the Ottawa and limestone sand mortars are approximately equal, but the average computed cement multiplier obtained from the river sand mortar is approximately 50 percent higher than the other two.

Table A5  
Mortar Test Series  
Al-Si Multipliers Obtained From Mortar Test

Water content - %	7.9	9.1	10.4	Avg
Al-Si Signature				
O.S.M.* Ottawa Sand	587.3	838.8	717.3	714.1
O.S.M. Cement	3243.9	2567.0	3030.0	2936.6
R.S.M.* River Sand	1578	2290	2474	2114
R.S.M. Cement	6124	3920	3690	4578
L.S.M.* Limestone Sand	890.0	880.8	435.0	735.3
L.S.M. Cement	2461.8	2661.1	4127.8	3083.5

\* O.S.M. = Ottawa Sand Mortar  
R.S.M. = River Sand Mortar  
L.S.M. = Limestone Sand Mortar

Table A6 lists the Al-Si multipliers obtained from dry constituent tests, constituent-water tests, and mortar tests. With the exception of the cement multiplier from the river sand mortars, there is general agreement between the multipliers obtained from the constituent-water tests and the mortar tests. This is true even though the water contents of the mortars were all below the water contents of the constituent-water tests used to compute the multipliers.

The three techniques described in the Ca-signature discussion were used to compute the Al-Si multipliers for the concrete test series. The average Al-Si multiplier obtained from the constituent-water data only used water contents greater than 13 percent.

Table A4 contains the Al-Si multipliers obtained from the three methods of computation. When counts per percent of concentration were used, there was reasonable agreement between the constituent-water

multipliers and the multipliers computed from the concrete mix data; however, when counts per weight concentration were used, all multipliers but one obtained from the constituent-water tests were greater than those computed from the concrete mixes.

Table A6  
Mortar Test Series  
Al-Si Multipliers - Percent Weight

Constituent	Dry Sample	Constituent/Water Test	Mortar Test	
		13.8 to 21.9% Water	O.S.M.*	
Cement (Table 5)	761	2556	2937	
% of Dry Sample	100	335	386	
		13.8 to 19.4% Water		
Ottawa Sand	804	837	714	
% of Dry Sample	100	104	89	
		16.7 to 21.9% Water	R.S.M.*	L.S.M.*
Cement (Table 7)	944	2994	4578	3084
% of Dry Sample	100	317	485	327
		10.7 to 16.7% Water		
River Sand	1569	2651	2114	
% of Dry Sample	100	169	135	
		10.7% H <sub>2</sub> O		
Limestone Sand	350	790	735	
% of Dry Sample	100	225	210	
* O.S.M. = Ottawa Sand Mortar				
R.S.M. = River Sand Mortar				
L.S.M. = Limestone Sand Mortar				

The theoretical accuracy (C.V. from counting statistics) obtained for the Al-Si signatures on the concrete test data varied from 0.33 to 0.22 percent for net peak varying from 273,000 to 135,000. The operational accuracy (C.V.) of the Al-Si signature varied between 1 and 2 percent for the repetitive tests on the three sets of filled polyester



samples. The associated counting statistics for the same samples averaged about 0.31 percent (Table A1). Thus, the operational accuracy of the Al-Si signature is considerably less than its upper bound counting accuracy, but when compared to the operational accuracy of the other signatures, it is approximately equal (Table A1).

In summary, the Al-Si signature (signature ratio intensity) is linearly related to the Al plus Si content of the test sample; however, since the signature sensitivity to the two different reactions is different and varies relative to the H (water) content of the sample, the resulting signature is complex and cannot be simply related to either Al or Si. The sensitivity of the Al-Si signature to elements other than H (water) appears to be negligible. The signature sensitivity to H is only significant when Al is present and when anhydrous samples are compared to samples containing water. For the variations in water content common to concretes and mortars, the signature's sensitivity to H is negligible. Due to the complex nature of the Al-Si signature and the H influence, there is no single or simple relationship between the Al-Si signatures for individual dry constituents (cement, fine aggregates, and coarse aggregates) and their associated signatures when combined with water. The theoretical error in the Al-Si signature for concrete type materials varied from 0.22 to 0.33 percent, and the operational error based on repetitive tests on a stable sample varied from 1 to 2 percent.

#### *C Signature*

The results of the previous study indicated that C signature intensities from mortars were lower than those obtained from dry constituent tests. Again it is assumed that the H in the mortar sample is acting as a neutron moderator. The  $^{12}\text{C}(n, n')^{12}\text{C}$  inelastic scattering reaction has a neutron energy threshold--4.43 MeV--that must be exceeded by the incident neutron if the reaction is to occur. Thus, increasing H contents increases the tendency for fast neutron moderation and, conversely, decreases the intensity of the C inelastic scattering reaction.

The net C peaks for the cement paste and ottawa sand data (Tables 5 and 6) are plotted relative to constituent content percent (Figure A28) and constituent content weight (Figure A29). Assuming that neither cement paste nor ottawa sand contain C, the C peaks associated with these two constituents and their mortar must be from the surrounding environment (background). It is assumed that the majority of the background C is coming from the WEP shielding materials. Both Figures A28 and A29 indicate significant fluctuation in the C signatures. Further linear regression equations relating constituent content percent to C signature indicate that the background carbon is influenced by the sample matrix and the constituent-water ratio of the sample, although the coefficients of determination indicate that the data fit is very poor ( $r^2 = .24/n$  for cement paste and  $r^2 = .03$  for ottawa sand-water).

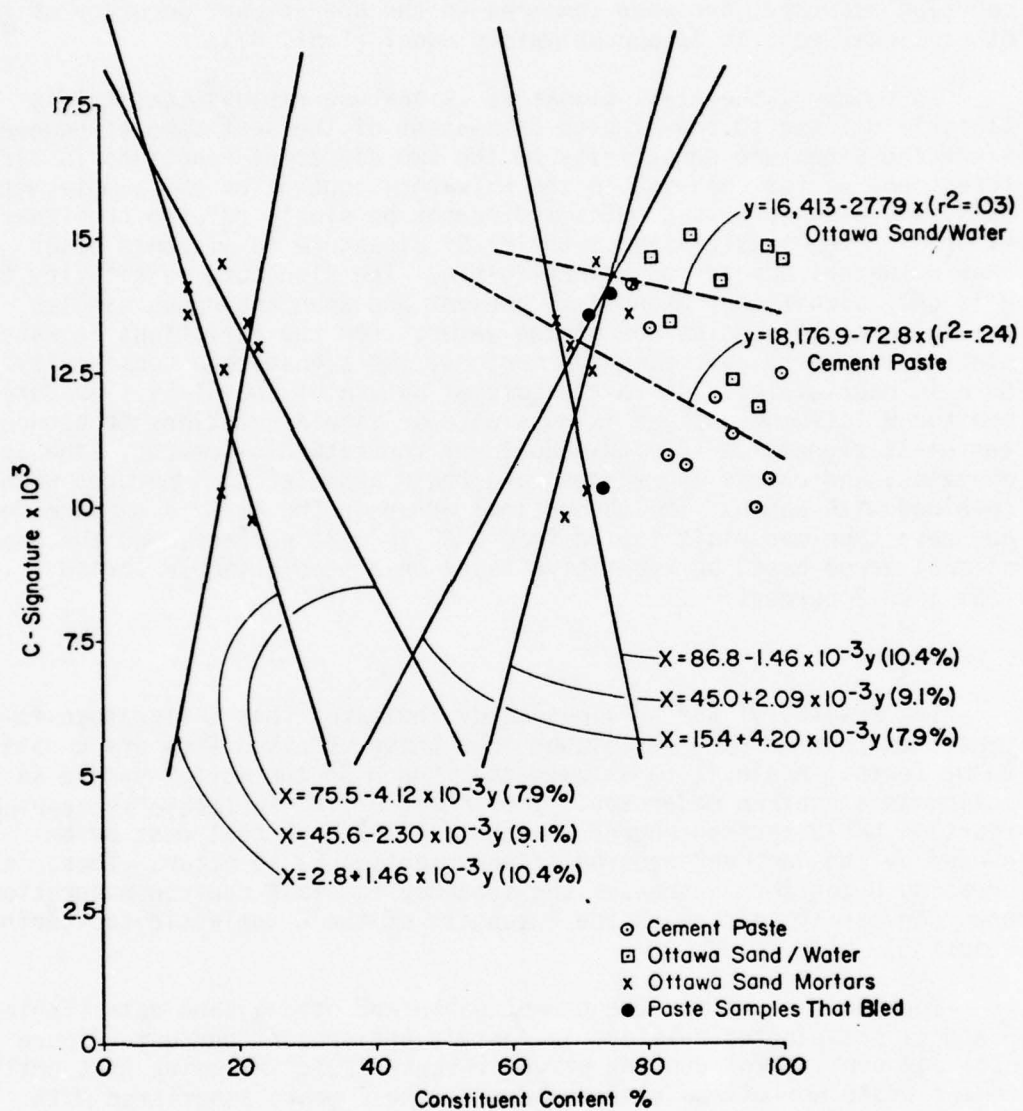


Figure A28. Mortar test series, C signature vs. constituent content - percent cement paste, ottawa sand/water, ottawa sand/mortar.

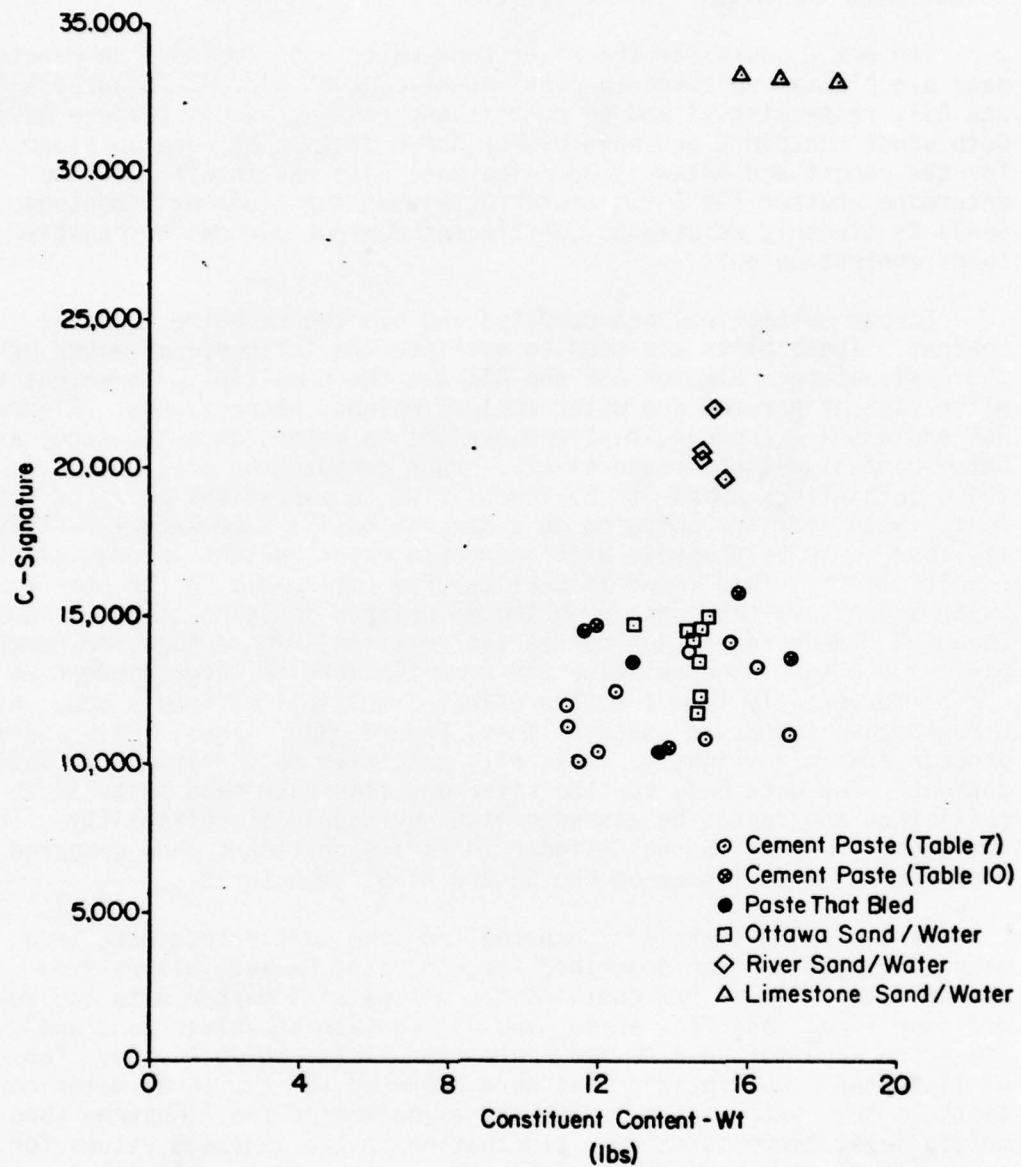


Figure A29. Mortar test series, C signature vs. constituent content - weight constituent/water data.

However, a simple comparison of the average of the C signatures for cement paste, 11,964 counts, and for ottawa sand-water, 13,891 counts, illustrates the significant difference.

The net C peaks for the river sand-water and limestone sand-water data are plotted relative to constituent content percent (Figures A30 and A31, respectively) and to constituent content weight (Figure A29). Both sands contain C and have higher net C peaks than were obtained for the cement and ottawa sand. The data base was insufficient to determine whether the C signature for either the river or limestone sands is linearly related to constituent content percent or constituent content weight.

Carbon multipliers are computed and plotted relative to water content. These plots are used to evaluate the influence of water on the C signature. Figures A32 and A33 are the C multiplier's weight to water content percent and water content weight, respectively. Figures A34 and A35 are the C multipliers percent to water content percent and water content weight, respectively. When computed on a weight basis, the C multipliers appear to be insensitive to variations in water content. When they are computed on a percent basis, some water sensitivity appears to be present, with increased water content increasing the C multipliers. This trend is particularly noticeable in the cement paste and ottawa sand data even though neither contains carbon. The computed linear regression curves (solid lines, Figure A34) for cement paste and ottawa sand relating the C multipliers to water content do not differ greatly from the theoretical C multipliers from a constant C background signature (dashed lines, Figure A34). Thus, the C backgrounds are only slightly, if at all, sensitive to variations in water content. The data base for the river and limestone sand tests is insufficient and cannot be assessed with any degree of reliability. The influence of water on the C signature is insignificant when compared with the water influence on the Ca and Al-Si signatures.

Carbon multipliers are computed from the mortar test data in a manner similar to that described for computing Ca multipliers from mortar data. Figure A28 contains the ottawa sand mortar data and regression lines, and Figures A30 and A31 contain the river sand and limestone sand mortar data and regression lines, respectively. Table A7 lists the C multipliers that were computed for the three water contents in the mortar tests. With the exception of the limestone sand multipliers, there is extreme fluctuation in the computed values for the multipliers. Very little information other than noting the magnitude of the fluctuations can be drawn from these computations.

Table A8 lists the C multipliers obtained from the dry constituents, constituent-water tests, and mortar tests. It is apparent that the C signature is relatively insensitive to water content; however, unlike the Ca and Al-Si signatures, there was basically no



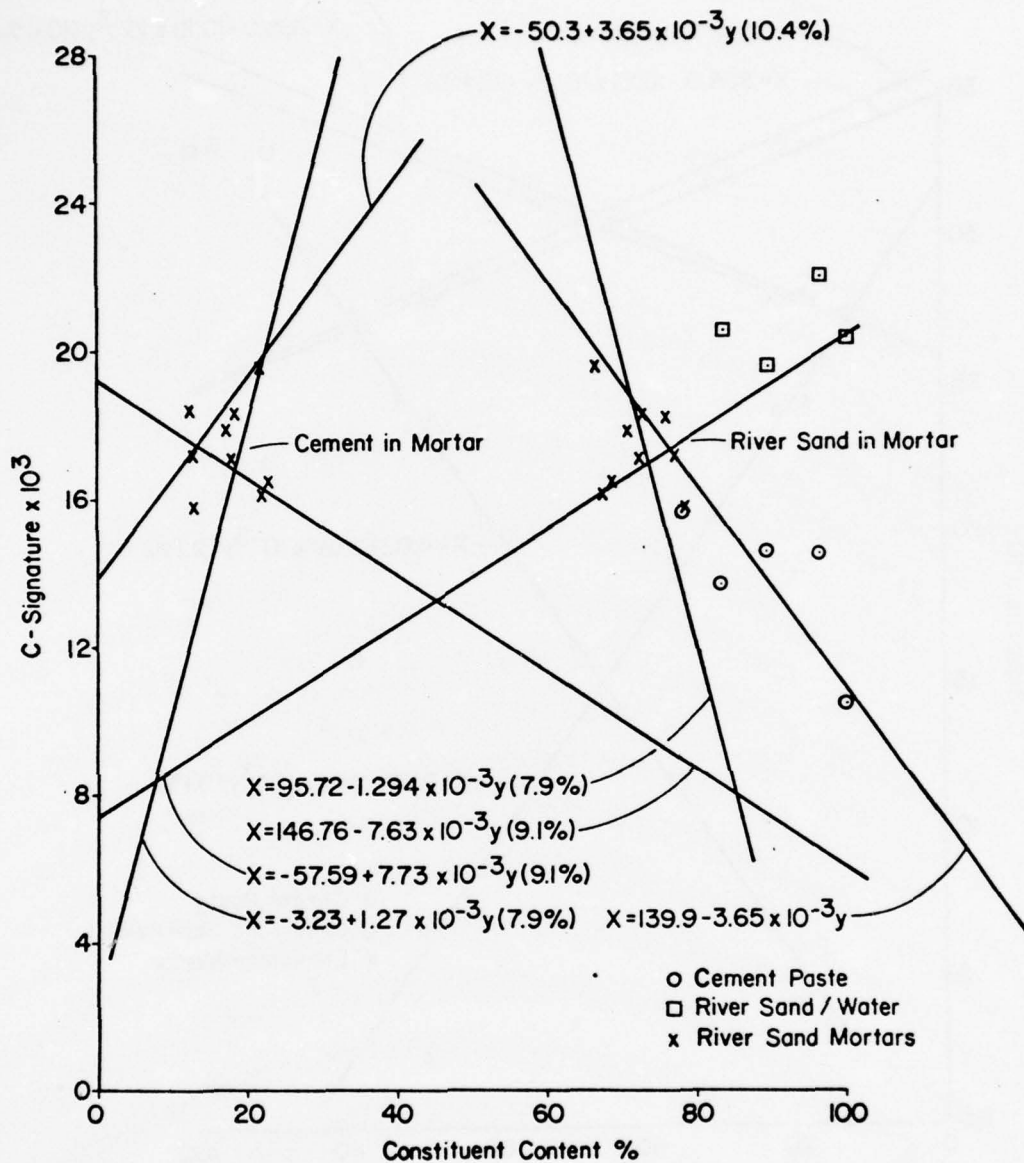


Figure A30. Mortar test series, C signature vs. constituent content - percent cement paste, river sand water, and river sand mortars.

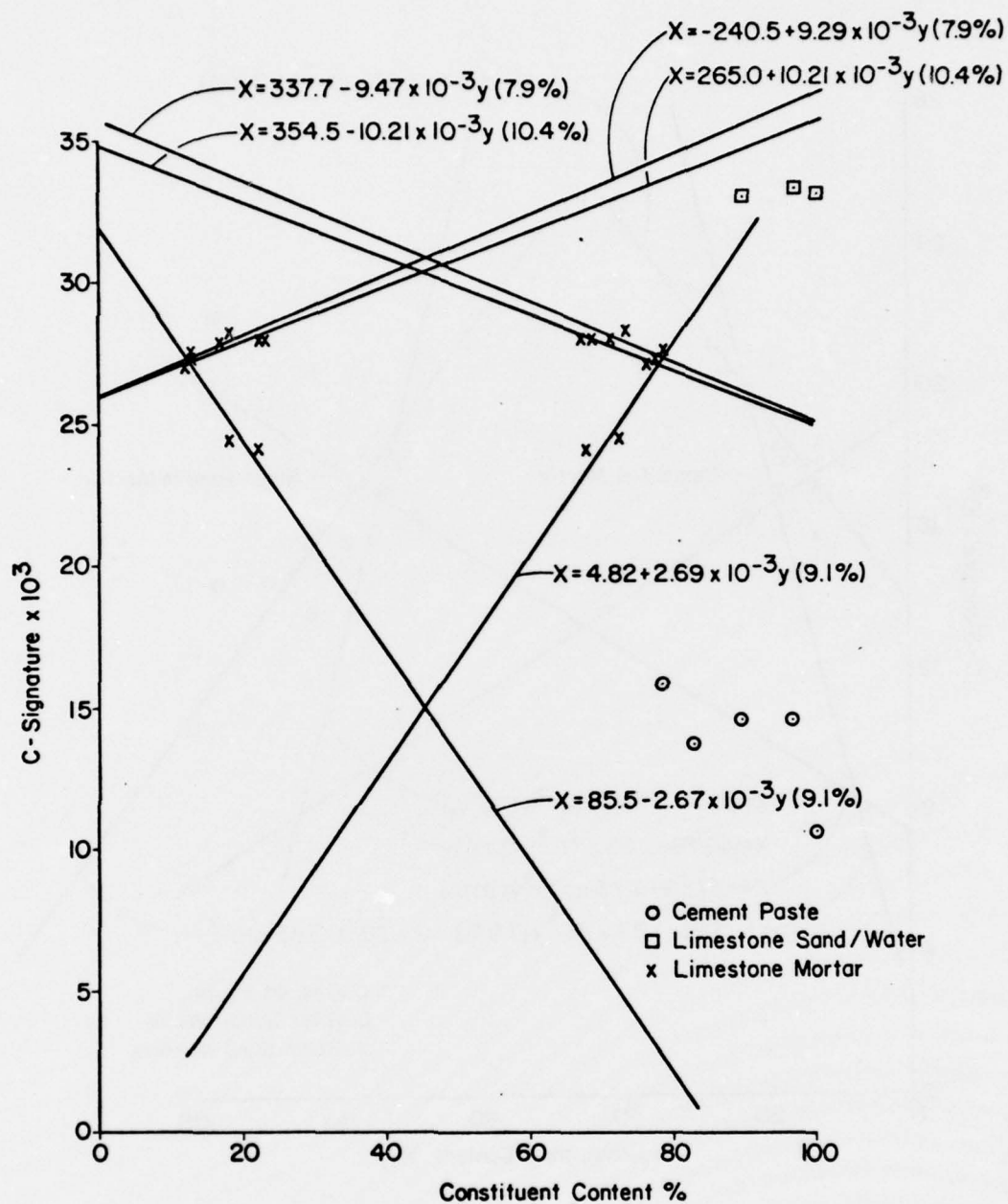


Figure A31. Mortar test series, C signature vs. constituent content - percent cement paste, limestone sand water, limestone sand mortars.

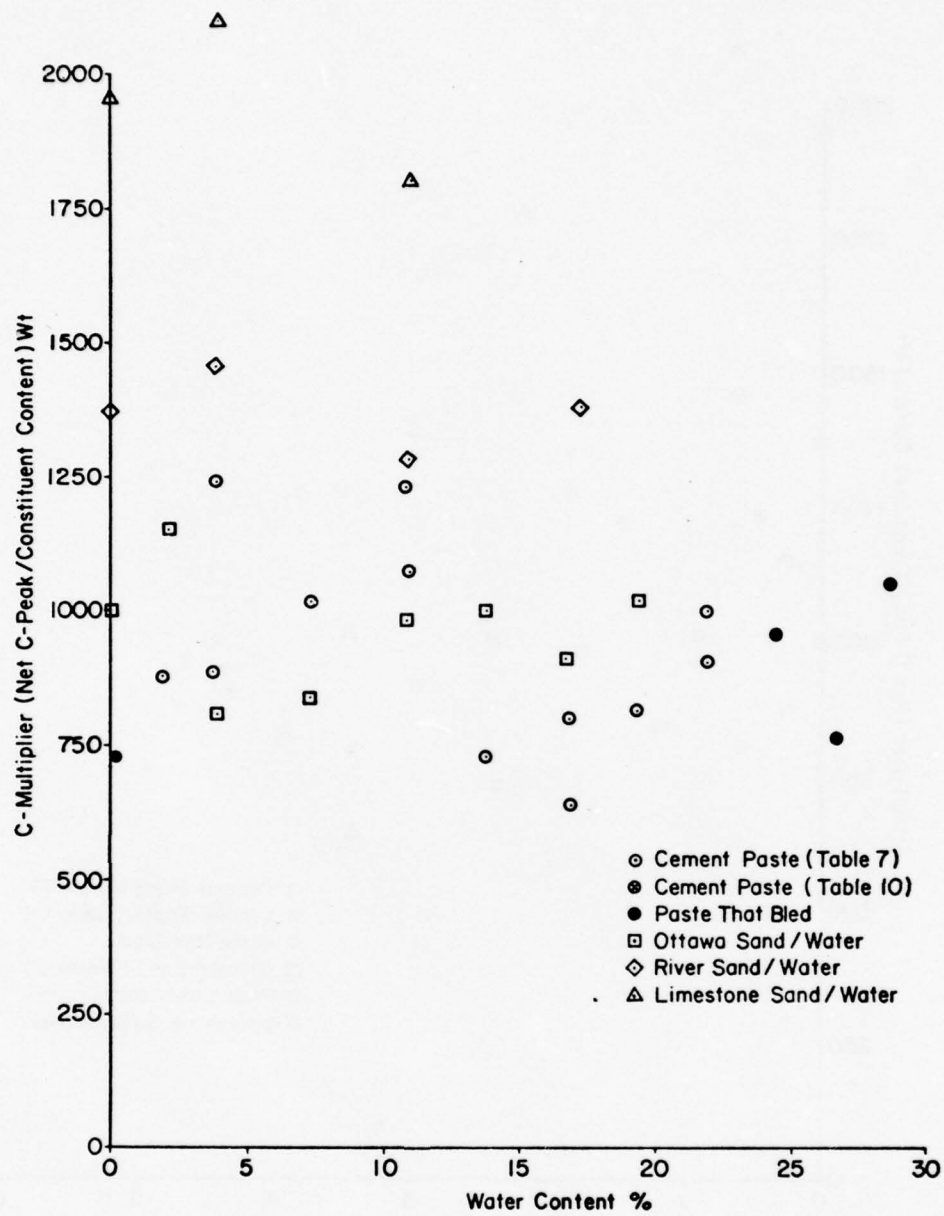


Figure A32. Mortar test series, C multiplier - weight vs. water content - percent constituent/water data.

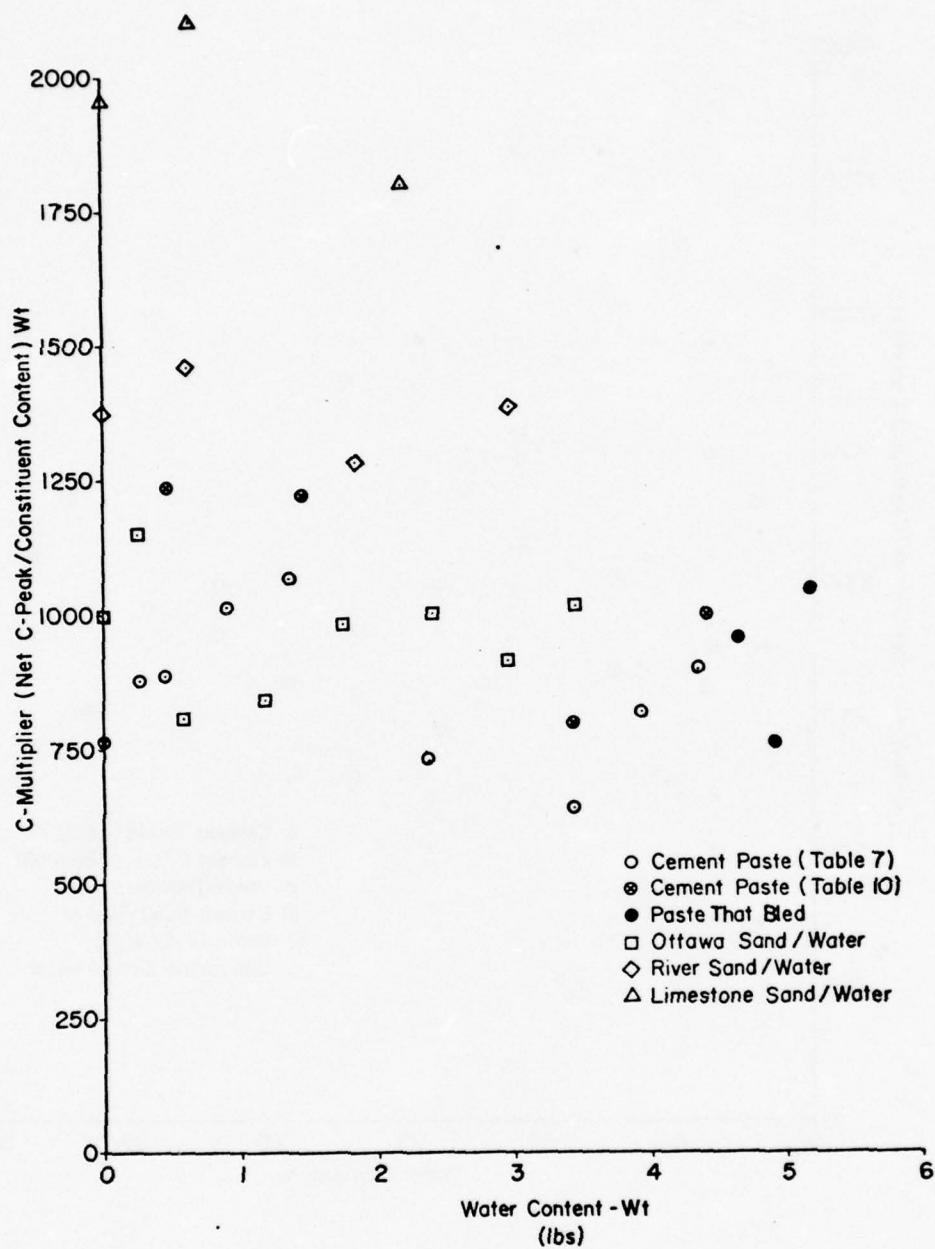


Figure A33. Mortar test series, C multiplier - weight vs. water content - weight constituent/water data.



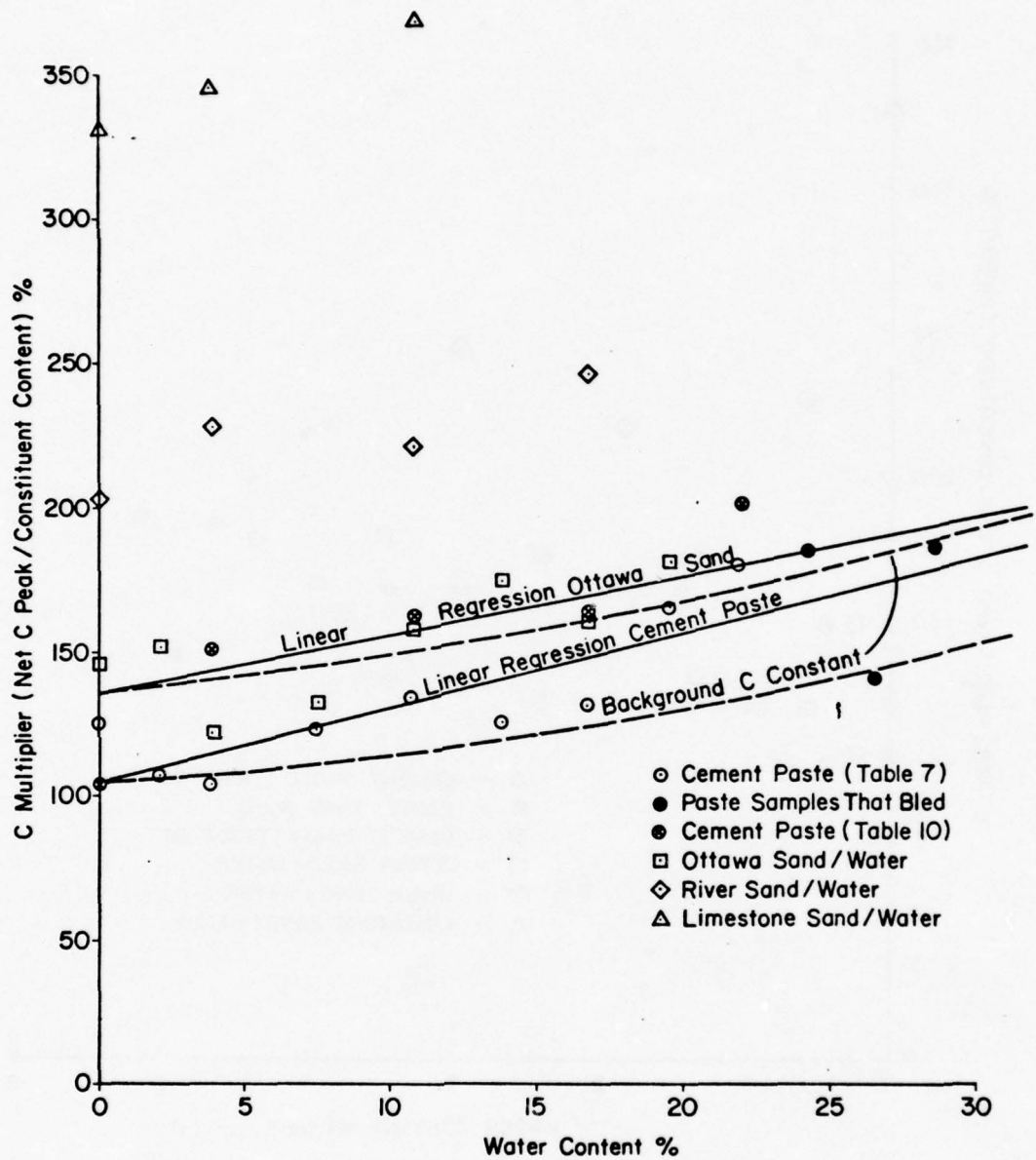


Figure A34. Mortar test series, C multiplier - percent vs. water content - percent constituent/water data.

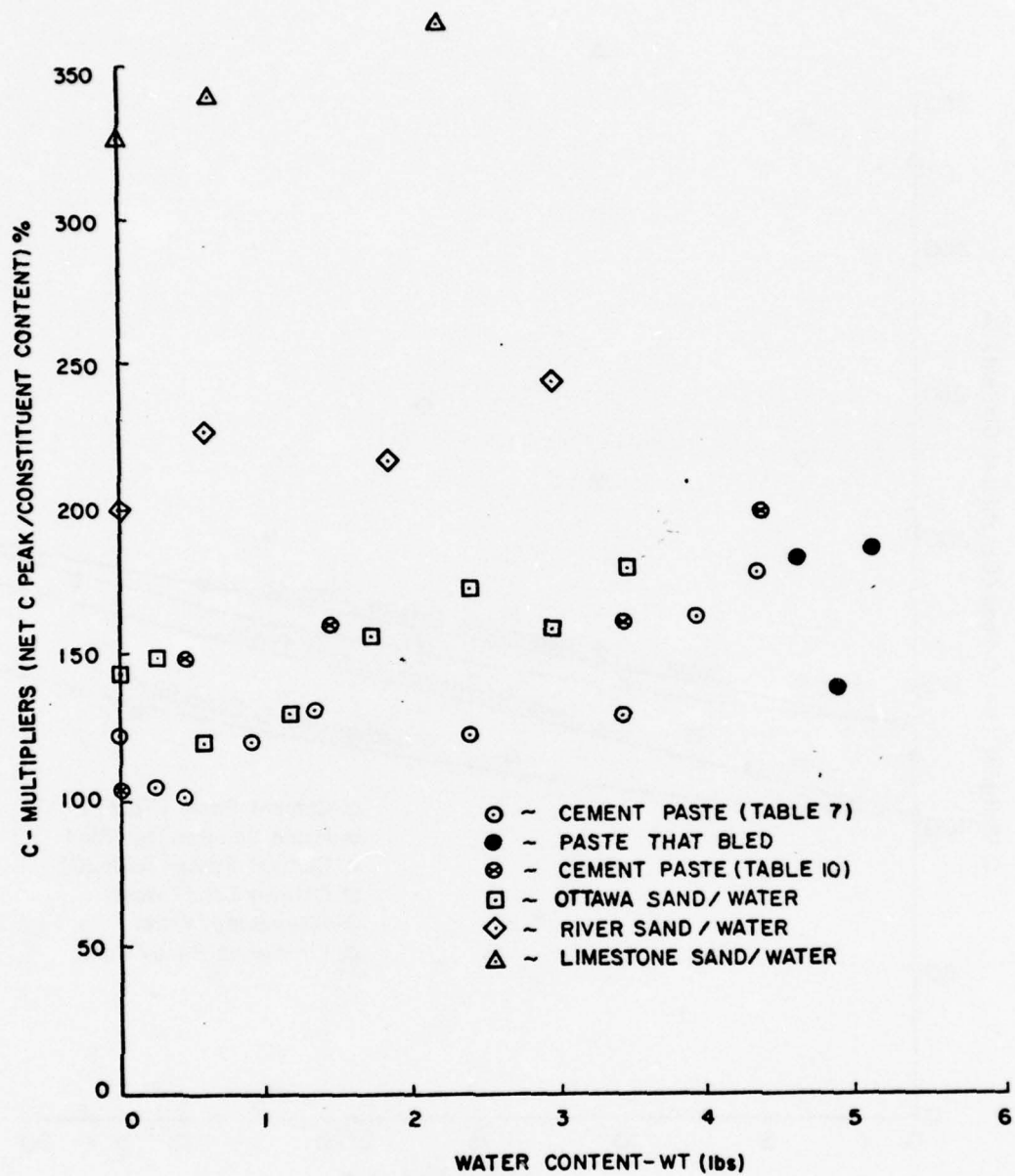


Figure A35. Mortar test series, C multiplier percent vs. water content - weight constituent/water data.

agreement between the C multipliers for constituent-water tests and mortar tests.

Table A7  
Mortar Test Series  
C Multipliers Obtained From Mortar Tests

Constituents	Water Content (%)			
C Signature	7.9	9.1	10.4	Avg
O.S.M.* Ottawa Sand	198.7	230.0	-21.8	155.3
O.S.M. Cement	-41.7	-226.6	663.6	65.3
R.S.M.* River Sand	29.1	211.5	153.7	131.4
R.S.M. Cement	808.8	81.3	427.5	439.2
L.S.M.* Limestone Sand	281.2	351.9	289.6	340.2
L.S.M. Cement	387.8	-21.0	387.6	251.1

\* O.S.M. = Ottawa Sand Mortar  
R.S.M. = River Sand Mortar  
L.S.M. = Limestone Sand Mortar

The C multipliers for the concrete data were computed by the three techniques used to compute the Ca and Al-Si multipliers for the same data. All levels of water content, including the anhydrous samples, were used to compute the C multipliers from the constituent-water test data. The average C peak from the cement paste constituent test was assumed to be the background C; this was subtracted (1) from the other signatures in the computations of the C multipliers from the constituent water data, and (2) from concrete data during the simple regression analysis.

The C multipliers computed from the concrete data agreed reasonably well with that obtained from the constituent data (Table A4), with the exception of the cement multipliers obtained from the cement-sand linear regression approach (method three). In these cases, the linear regression curves indicated that increasing cement contents and decreasing sand contents produced increased C signatures. This is really a physical impossibility, but was probably a result of the poor accuracy or sensitivity of the C signature.

Using counting statistics, the theoretical accuracy (C.V.) obtained from the C signature on the concrete test data varied from 5.93 to 3.59 percent for net counts varying between 12,500 and 20,000 (27.4 and 44.0 counts/PHA time); however, these accuracies are deceptively

Table A8  
Mortar Test Series  
C Multipliers - Percent Weight

Constituent	Dry Sample	Constituent/Water	Mortar Test	
		13.8 to 21.9% H <sub>2</sub> O	(Avg) *	
			O.S.M.	
Cement (Table 5)	125.1	150.8	65.3	
% of Dry Sample	100.0	120.5	52.2	
		13.8 to 19.4% H <sub>2</sub> O		
Ottawa Sand	146.7	172.6	155.3	
% of Dry Sample	100.0	117.6	105.9	
		16.7 to 21.9% H <sub>2</sub> O	R.S.M. *	L.S.M. *
Cement (Table 7)	105.1	182.8	439.2	251.5
% of Dry Sample	100.0	174.0	418.0	240.0
		10.7 to 16.7% H <sub>2</sub> O		
River Sand	203.7	233.6	131.4	
% of Dry Sample	100.0	115.0	64.5	
		10.7% H <sub>2</sub> O		
Limestone Sand	331.0	369.6	340.2	
% of Dry Sample	100.0	111.7	102.8	

\* O.S.M. = Ottawa Sand Mortar  
R.S.M. = River Sand Mortar  
L.S.M. = Limestone Sand Mortar

high, since it can be assumed from the constituent-water test on cement (which contains no C but has a net C signature of approximately 23 counts per second) that 23 counts per second is associated with the C background of the WEP shielding material. Thus, the actual counting accuracy should be based on the values obtained minus the 23 counts per second background. If this is done, the counting accuracies (C.V.) vary from 7.6 to 40 percent. The operational accuracy (C.V.) of the C signature varied between 1.5 and 5 percent for the repetitive tests on the three sets of polyester concrete samples (Table A1). The associated counting statistics when the background was not removed was approximately 2.1 percent, and with the background removed, 3.0 percent;



this indicated reasonable correlation between the upper bound counting statistics and the actual sampling statistics, although in both cases, the accuracy level was very poor.

In summary, two dominant factors can be observed from the C-signature analysis: (1) a large percentage of the C signature is from the surrounding media and is not associated with the sample being tested; and (2) if the background C is removed from the net signature, the theoretical accuracy of the C signature for concrete-type materials ranges from 7.6 to 40 percent. This level of accuracy is unacceptably low and makes further analysis of the C signature nearly meaningless.

### *Si Signature*

The results of the previous study indicated that Si-signature intensities from mortars were lower than those obtained from dry constituent tests. Again, as with the C, it is assumed that the H in the mortar acts as a neutron moderator. The  $^{28}\text{Si}(n, n')^{28}\text{Si}$  inelastic scattering reaction has a neutron energy threshold of 1.78 MeV, which must be exceeded by the incident neutrons if the inelastic scattering reaction is to occur. Thus, increasing H contents increases the tendency for fast neutron moderation, conversely decreasing the intensity of the Si inelastic scattering reaction.

The net Si peaks for the cement paste and ottawa sand data (Tables 5 and 6) are plotted relative to constituent content percent (Figure A36) and constituent content weight (Figure A37). Comparing the two figures indicates that the Si signature is nearly linearly related to constituent content percent. Conversely, no relationship is apparent between the Si signature and constituent content weight. The net Si peaks for the river sand-water and limestone sand-water data are plotted relative to constituent content percent (Figure A38 and A39, respectively) and to constituent content weight (Figure A37). Although the quantity of data is much smaller, the same basic trend is observed for the river sand-water data. The limestone sand-water data were insufficient (two tests) to draw any conclusions.

To further evaluate these trends, Si multipliers were computed and plotted relative to water content. Figures A40 and A41 are the Si multipliers' weight to water content percent and water content weight, respectively. Figures A42 and A43 are the Si multipliers' percent to water content percent and water content weight, respectively. When computed on a weight basis, the Si multipliers appear to be water-content-sensitive, and decreased by increasing water contents. However, when computed on a percent basis, the multipliers appear to be reasonably insensitive to water content. This is clearly evident in the cement paste data and only slightly less in the ottawa and river sand data.

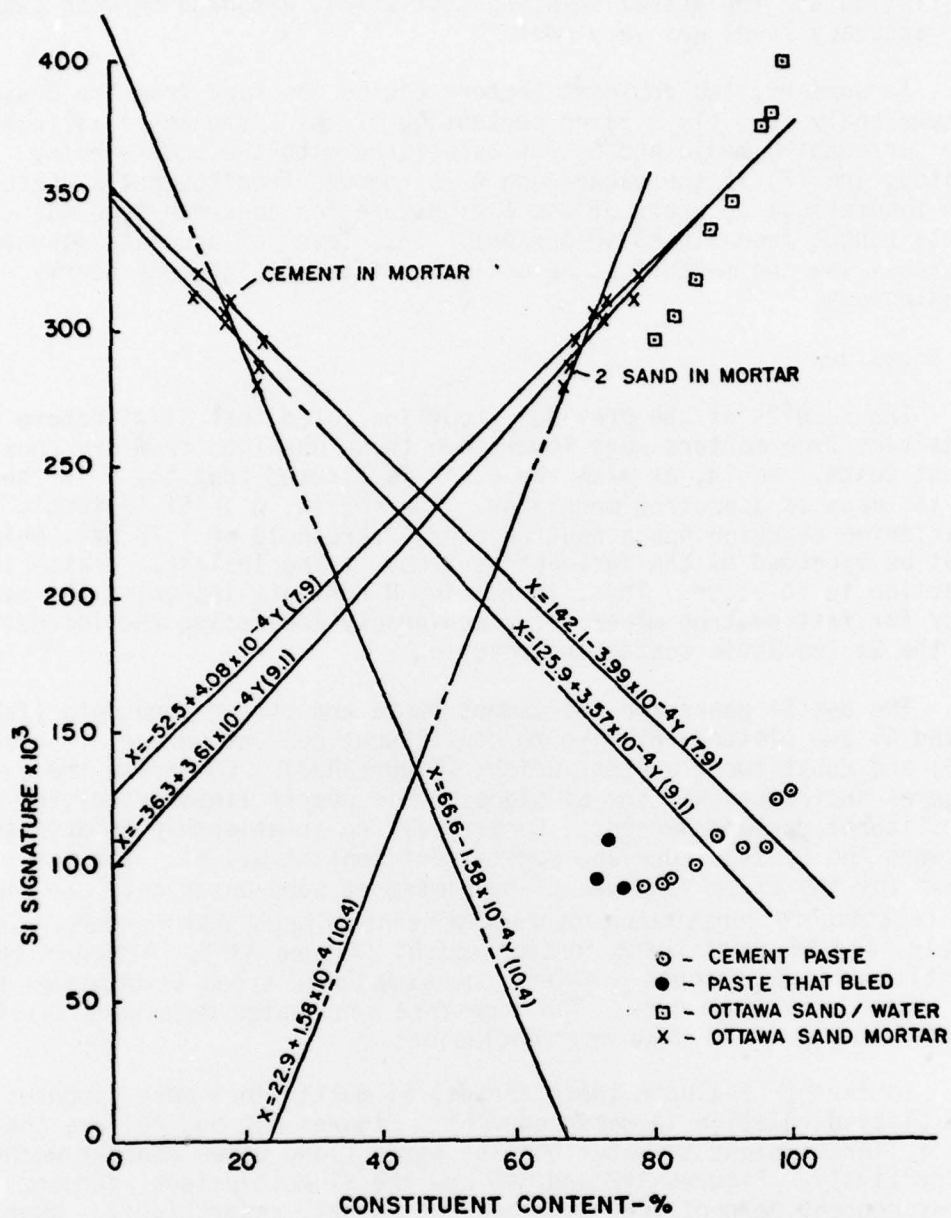


Figure A36. Mortar test series, Si signature vs. constituent content - percent cement paste, ottawa sand water, ottawa sand mortar.

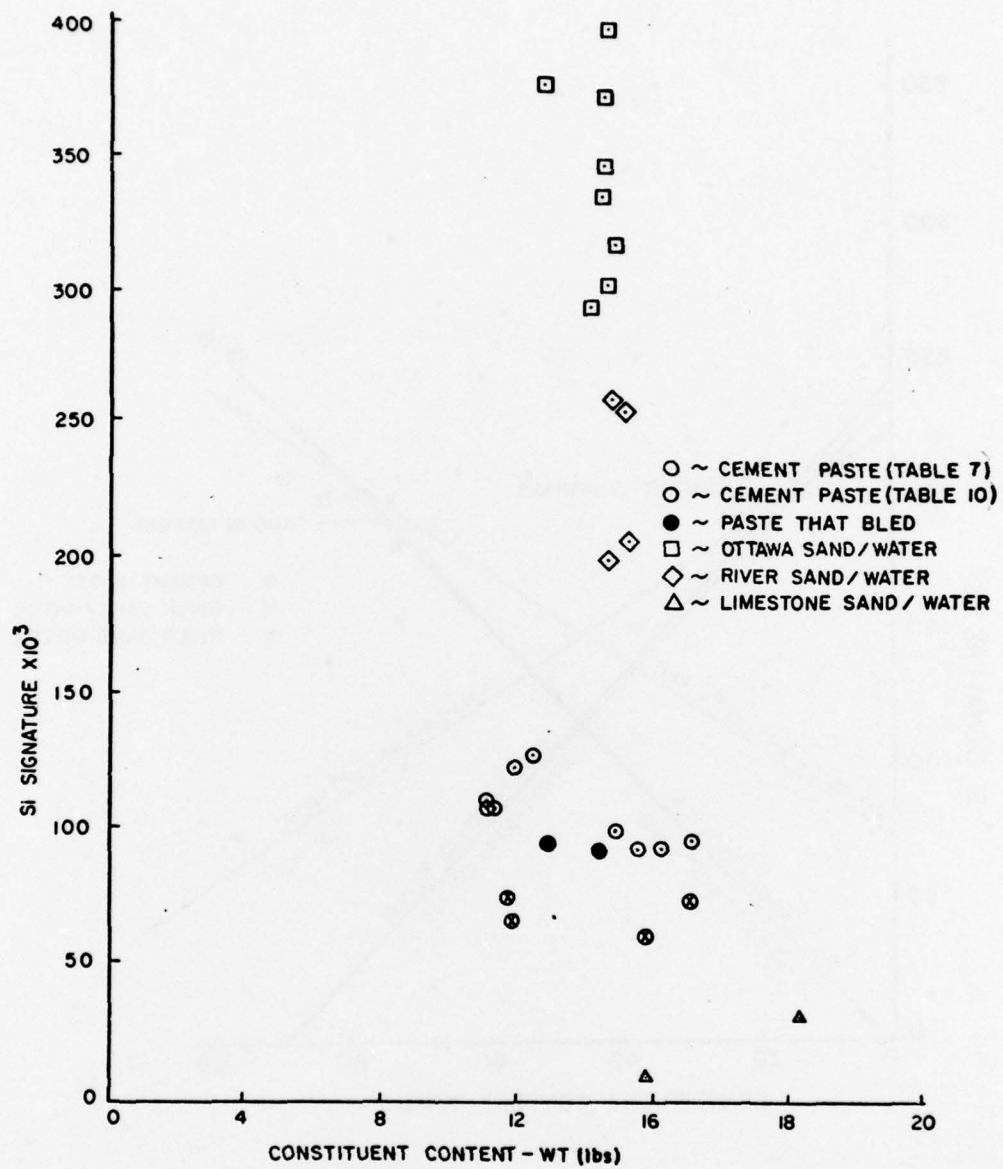


Figure A37. Mortar test series, Si signature vs. constituent content - weight constituent/water data.

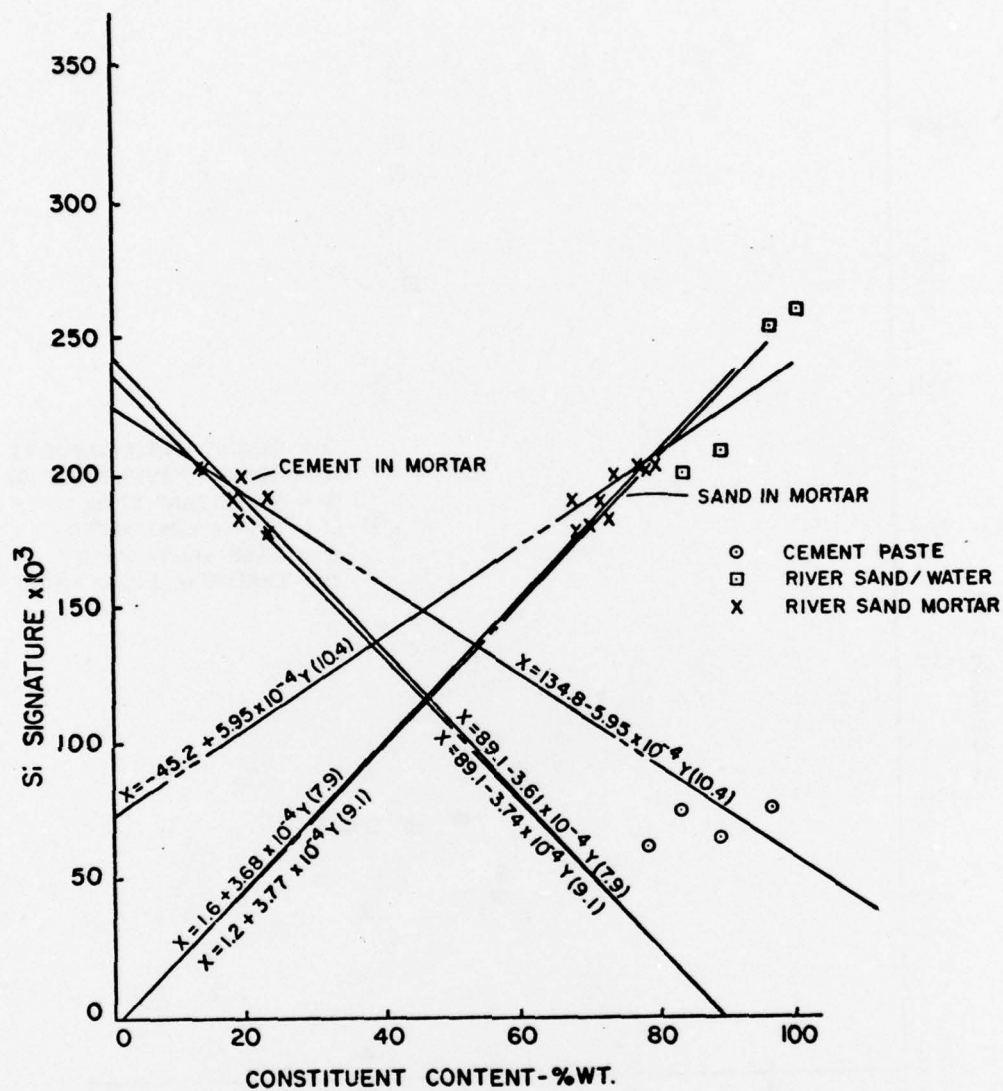


Figure A38. Mortar test series, Si signature vs. constituent content - percent weight cement paste, river sand water, river sand mortar.



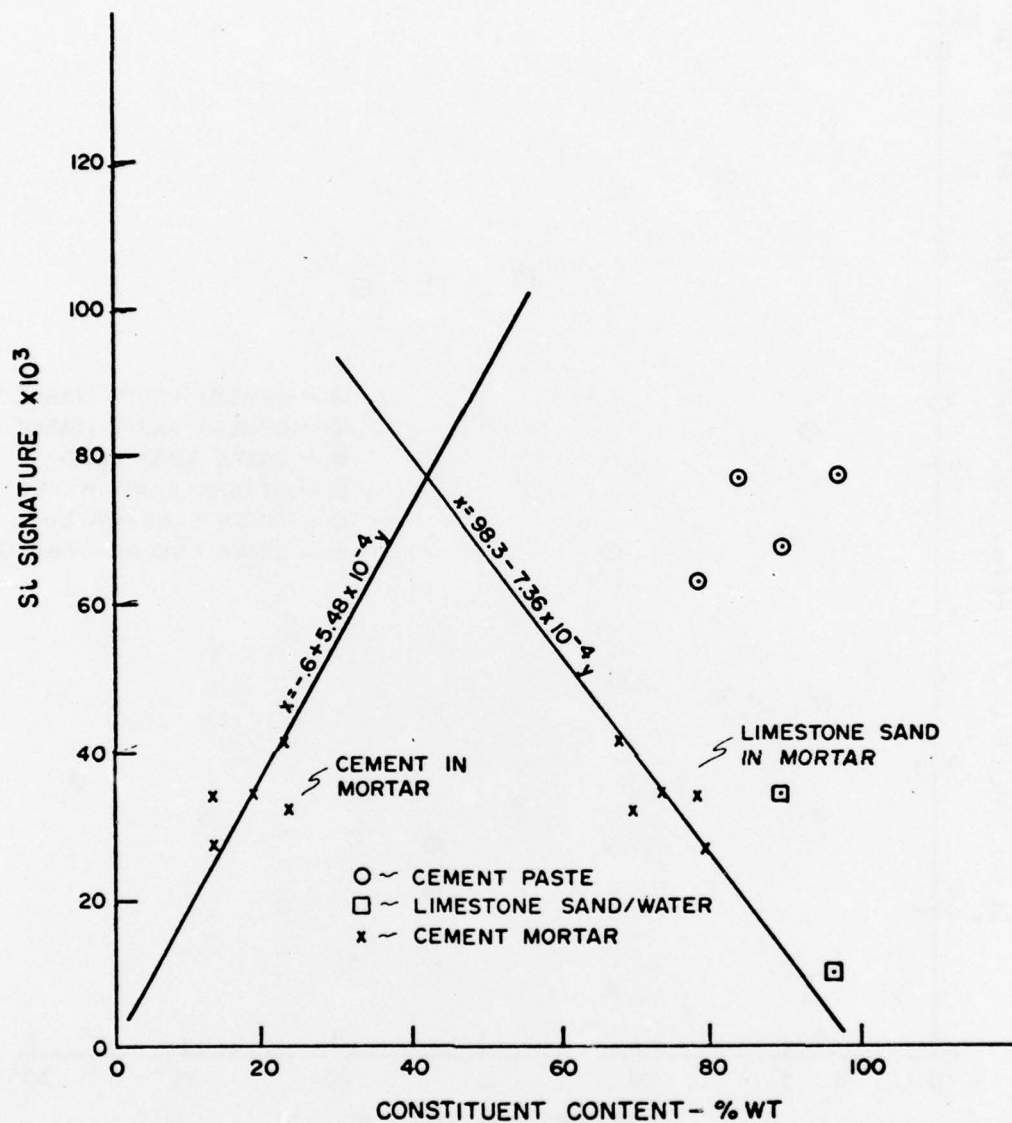


Figure A39. Mortar test series, Si signature vs. constituent content - percent weight cement paste, limestone sand water, limestone sand mortar.

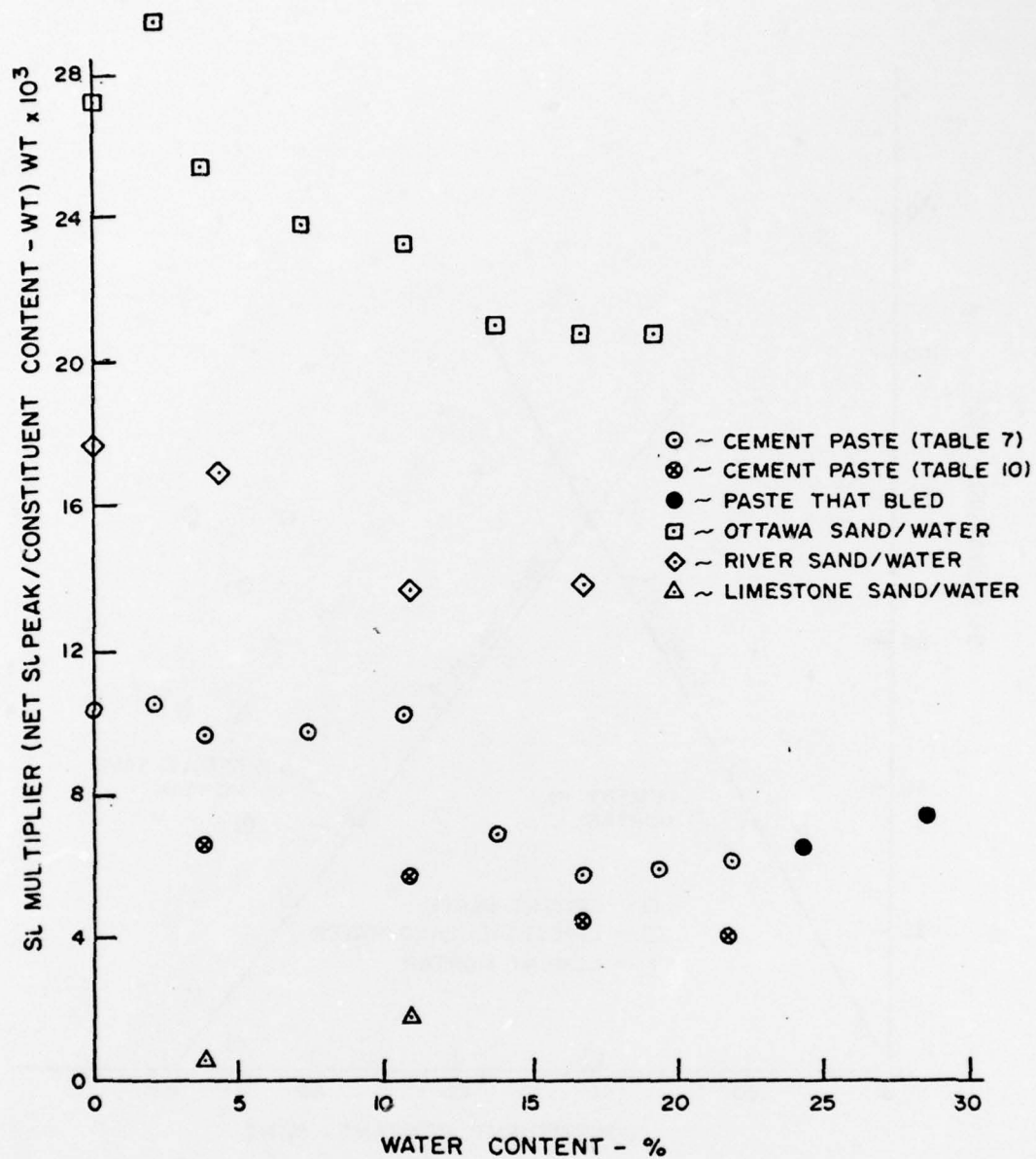


Figure A40. Mortar test series, Si multiplier - weight vs. water content - percent constituent/water data.

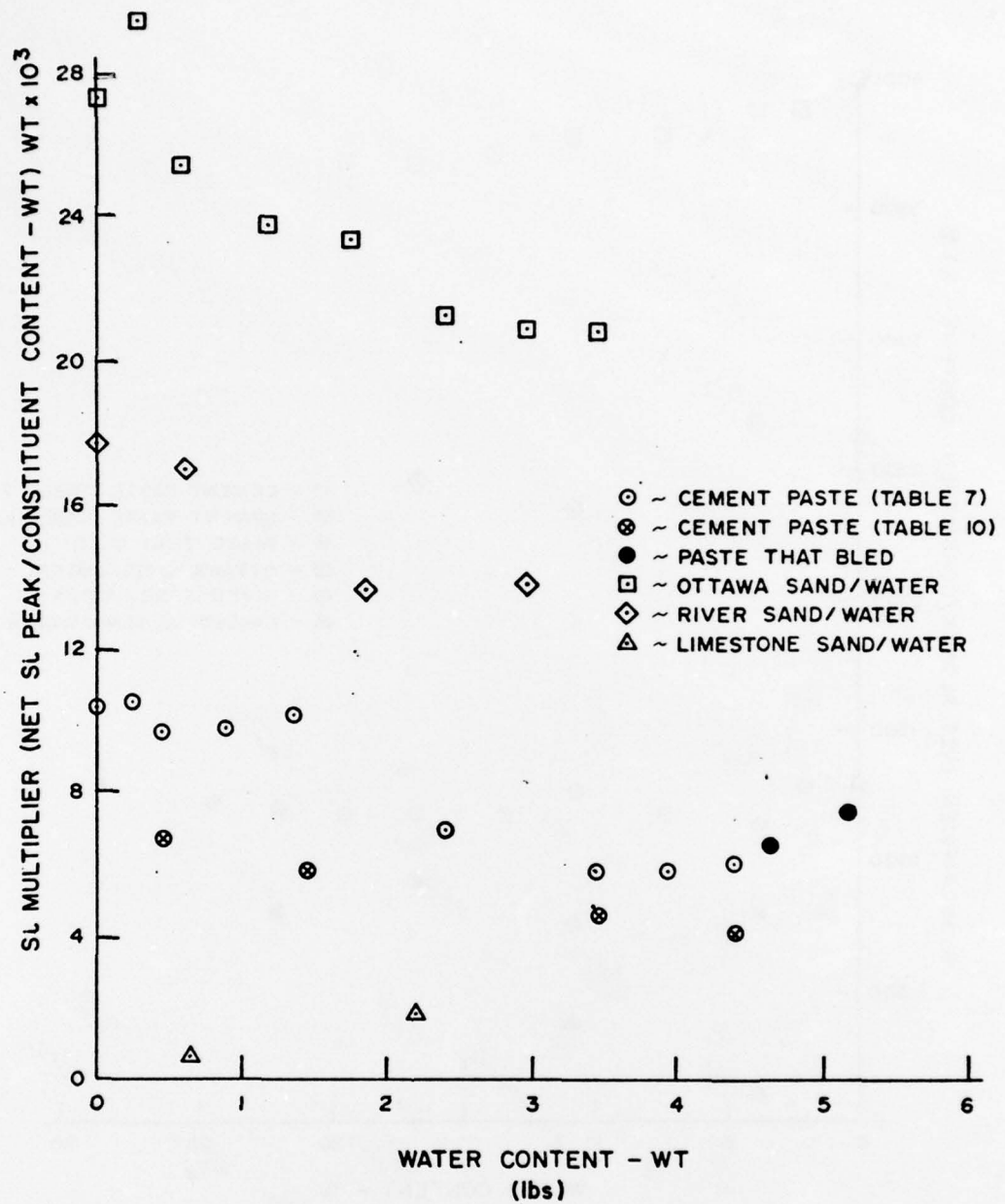


Figure A41. Mortar test series, Si multiplier - weight vs. water content - weight constituent/water data.

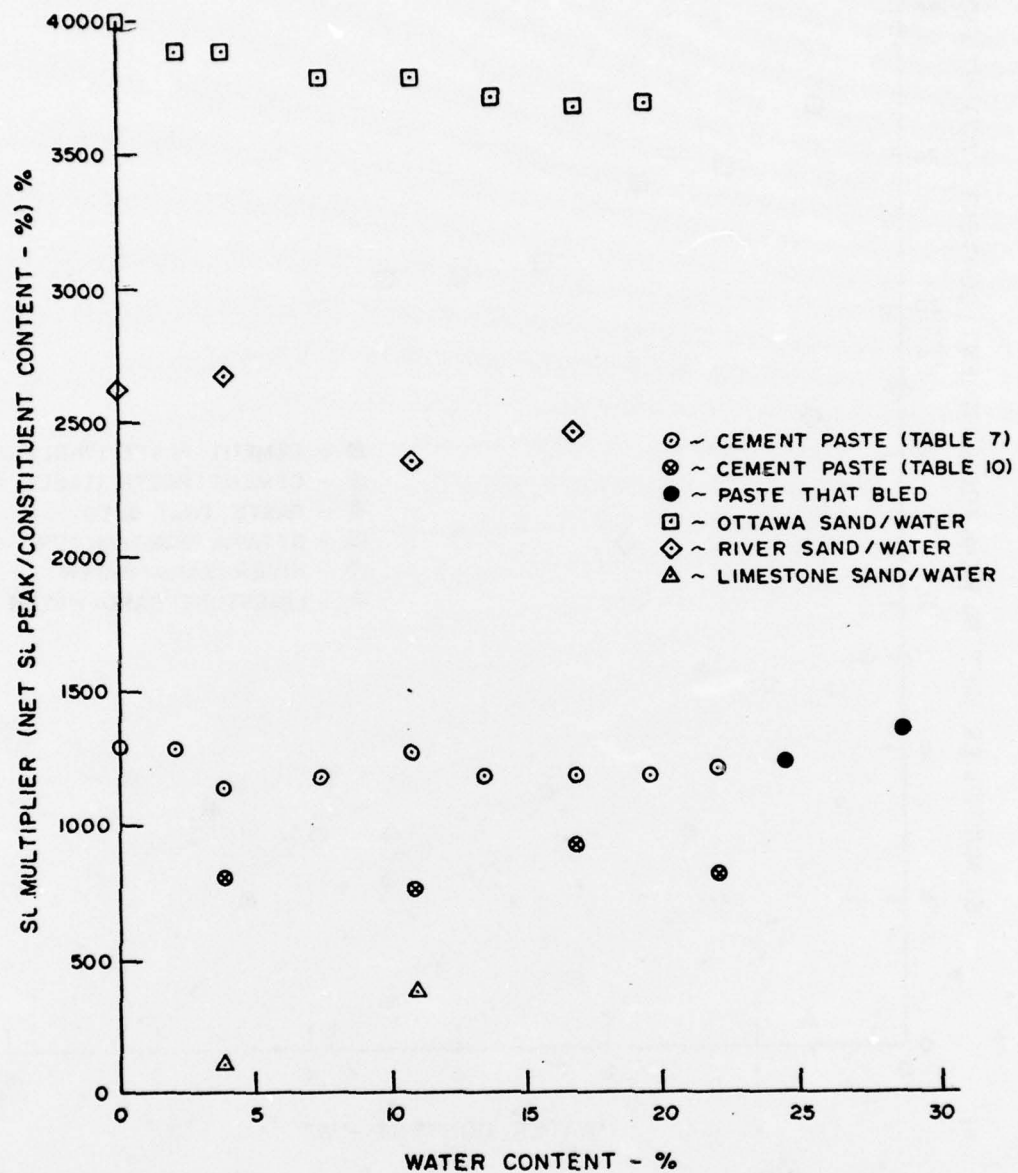


Figure A42. Mortar test series, Si multiplier - percent vs. constituent content - percent constituent/water data.



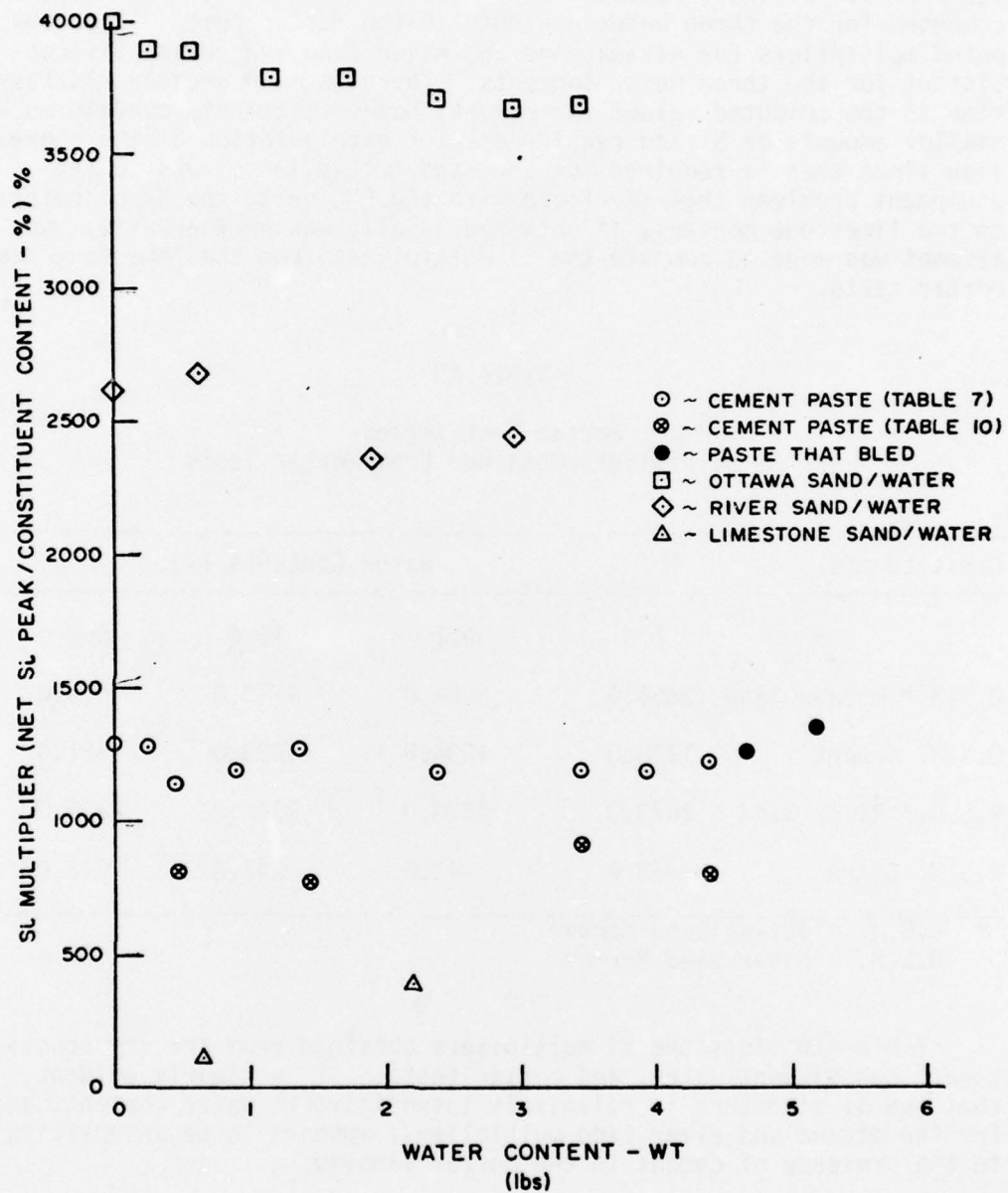


Figure A43. Mortar test series, Si multiplier - percent vs. water content - weight constituent/water data.

Silicon multipliers are computed for the Ottawa sand and river sand mortar data by the method described for computing Ca multipliers. Figures A36 and A38 contain the Ottawa sand and river sand mortar data and regression lines, respectively. Table A9 lists the Si multipliers computed for the three water contents in the mortar tests. The computed multipliers for Ottawa sand and river sand are reasonably consistent for the three water contents. There is much greater fluctuation in the computed values for cement; however, cements contain much smaller amounts of Si and require greater extrapolation of the regression lines than is required for the sand multipliers. Due to the equipment problems that developed with the FNC unit, the Si signature on the limestone mortars, if obtained at all, was very erratic. No attempt was made to compute the Si multipliers from the limestone sand mortar tests.

Table A9  
Mortar Test Series  
Si Multipliers Obtained From Mortar Tests

Constituents	Water Contents (%)			
	7.9	9.1	10.4	Avg
O.S.M.* Ottawa Sand	3856.0	3881.0	4710.0	4079.0
O.S.M. Cement	1378.0	1094.0	-1623.0	521.0
R.S.M.* River Sand	2673.0	2623.0	2528.0	2608.0
R.S.M. Cement	-68.4	-43.2	847.0	245.0

\* O.S.M. - Ottawa Sand Mortar  
R.S.M. - River Sand Mortar

Table A10 lists the Si multipliers obtained from the dry constituent, constituent-water, and mortar tests. It is clearly evident that the Si signature is relatively insensitive to water content, and for the Ottawa and river sand multipliers, appears to be insensitive to the presence of cement in the mortar samples.

The three techniques previously described in the concrete data analysis were also used to compute the Si multipliers. Both the dry-constituent and constituent-water data were used to compute the multipliers from the constituent data.

Table A10  
Mortar Test Series  
Si Multipliers - Percent Weight

Constituent	Dry Sample	Constituent/Water	Mortar Test
		12.8 to 21.9% H <sub>2</sub> O	(Avg)
Cement (Table 7)	1298	1195	521
% of Dry Sample	100	92	40
		13.8 to 19.4% H <sub>2</sub> O	
Ottawa Sand	4006	3691	4079
% of Dry Sample	100	92	102
		16.7 and 21.9% H <sub>2</sub> O	
Cement (Table 10)	804*	861	245
% of Dry Sample	100	107	30
		16.7% H <sub>2</sub> O	
River Sand	2623	2444	2608
% of Dry Sample	100	93	99

\* 3.85 percent water in sample

Table A4 contains the Si multipliers obtained from the three methods of computation. The three methods of computation agree reasonably well for the multipliers computed on a percent basis; however, when the multipliers were computed on a weight basis, the constituent water data produced larger multipliers than those computed by multiple linear regression from the concrete data.

The theoretical accuracy (C.V. from counting statistics) obtained from the Si signature on the concrete test data varied from 0.77 to 2.29 percent for net counts ranging from 211,000 to 72,000 (460 to 157 counts per second). The operational accuracy (C.V.) based on the three sets of repetitive tests on the polyester concrete samples varied from 3.7 to 1.34 percent, while the associated counting statistics (C.V.) for the samples averaged about 1 percent (Table A1). Thus, the operational error was about twice that of the upper bound counting error

In summary, the data indicate that the Si signature is linearly related to the SI content of the sample. The Si signature is not

sensitive to other elements common to concrete. On a percentage constituent basis, there appears to be a direct correlation between the individual dry constituents and their associated signatures when combined with water. The theoretical error in the Si signature varied from 0.77 to 2.29 percent, and the operational error based on repetitive tests of a given sample varied from 1.34 to 3.2 percent.



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